

# The Journal *of the* SOCIETY OF DYERS AND COLOURISTS

Volume 72      Number 8

## CONTENTS

|  |     |
|--|-----|
| PUBLICATIONS SPONSORED BY THE SOCIETY'S FASTNESS<br>TESTS CO-ORDINATING COMMITTEE  |     |
| XIX—RECOMMENDED CONDITIONS FOR FADING LAMP OPERATION<br>( <i>Light Fastness Subcommittee</i> )   | 369 |
| COMMUNICATIONS   |     |
| THE INFLUENCE OF HISTOLOGICAL FACTORS ON THE<br>ATTACK OF THE WOOL FIBRE BY VARIOUS REAGENTS<br>( <i>R. L. Elliott and J. B. Roberts</i> )   | 370 |
| A STUDY OF THE PHOTOCHEMISTRY OF DYES ON PROTEINS AND<br>OTHER SUBSTRATES<br>( <i>J. W. Cumming, C. H. Gilas, and Miss A. E. McEachran</i> ) | 373 |
| ERRATA   | 381 |
| NOTES  | 382 |
| OBITUARY NOTICE  | 383 |
| NEW BOOKS AND PUBLICATIONS   | 383 |
| ABSTRACTS  | 384 |

THE SOCIETY OF DYERS AND COLOURISTS  
19 PICCADILLY · BRADFORD 1 · YORKSHIRE

## Notice to Authors of Papers

The MSS. of all papers communicated to or read before the Society become the property of the Society. They should be addressed to the Editor, Society of Dyers and Colourists, 19 Piccadilly, Bradford 1. Authors must not allow their papers to be published elsewhere before they have appeared in the Society's *Journal*. Should prior publication take place without the sanction of the Publications Committee, the paper will be printed only as an abstract or summary.

Manuscripts submitted for publication in the *Journal* should be typewritten (double spacing) on good-quality paper, using one side of the paper only and leaving a margin at least 1 in. wide on the left-hand side. The time taken in refereeing papers (both lectures and communications) will be reduced to a minimum when authors submit two copies of the typescript.

In view of the high costs of publication, it is essential that authors should be as concise as possible. When experimental procedure has already been published, a literature reference to the paper containing the details is sufficient, whilst well known experimental methods should be described very briefly.

Introductory paragraphs describing the aims of the investigation and the method of attack are desirable, and should be followed by the experimental results and their discussion. There should be a brief summary for insertion at the beginning of the paper. References to the literature should be numbered consecutively, using superscript numbers without brackets immediately following the text words or author's name to which they refer.

The list of references should be given at the end of the manuscript and the abbreviations used should be, as far as possible, those given in the "List of Periodicals Abstracted" included at the end of the Index to the preceding year's *Journal*. Reference numbers in this list should be neither enclosed in brackets nor followed by full-stops. As far as possible throughout the manuscript the abbreviations listed in the Jan. 1950 issue (p. 54) should be used. Tables should be numbered consecutively in Roman numerals and Figures in Arabic numerals.

The number of figures and graphs should be kept as low as possible, and data should be presented in the form of either tables or graphs, not both. Drawings should be carefully prepared, preferably in Indian ink, on plain white drawing paper or, preferably, Bristol board. In graphs, the frame and actual curves should be ruled and inked more heavily than any co-ordinate lines, and the latter should not be close together as in ordinary graph paper. Experimental points should always be given, and where several graphs appear in a single Figure clear means of differentiation must be adopted. All numbers and legends are set up in type by the printer, and authors should therefore indicate them lightly in pencil.

Twenty-five free copies of a reprint are supplied to the author of an original paper published in the *Journal*, or fifty free copies are supplied when there are two or more authors, and a further number may be purchased from the Society at the rates given below.

### REPRINTS OF LECTURES AND COMMUNICATIONS

Reprints of all lectures and communications are available after publication to members and non-members of the Society. The charges (postage included) are as follows—Single copies 2s. 6d. each; per dozen copies up to and including 8 pages, 12s. 6d., and for papers occupying more than 8 pages of the *Journal*, 17s. Orders should be addressed to "The Society of Dyers and Colourists, 19 Piccadilly, Bradford 1". They can be accepted only if accompanied by remittance and if received immediately after publication of the paper.

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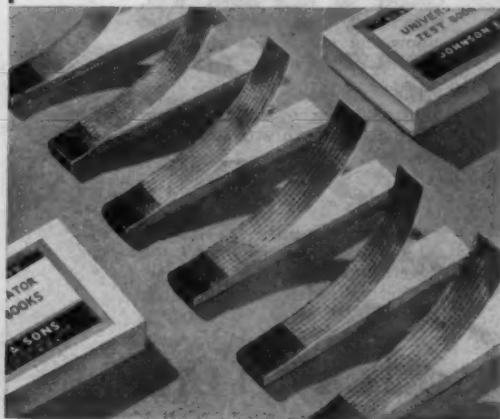
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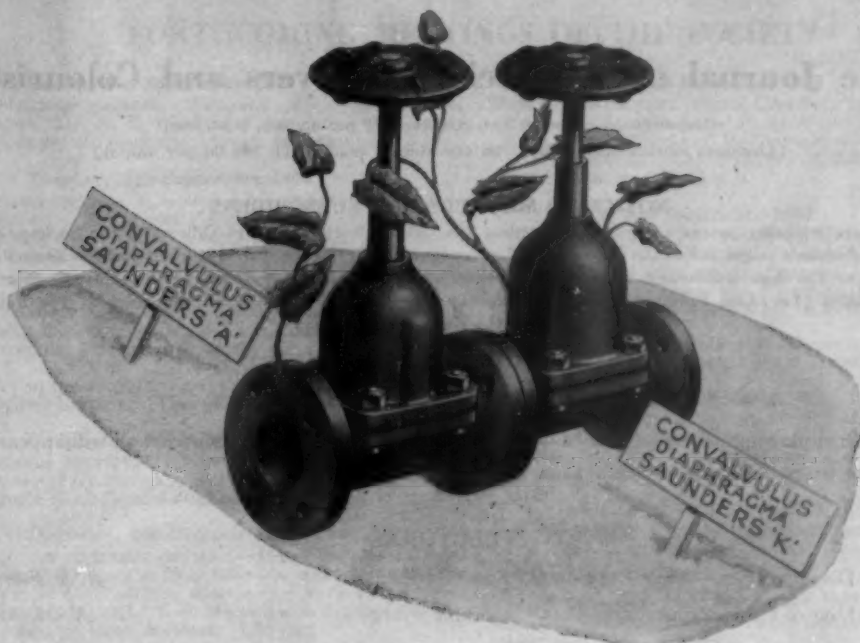
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### NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring *general information* regarding the Official Notices, List of Officers of the Society, etc. should consult pages 1-6 of the January 1956 and pages 313-317 of the July 1956 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire (Telephone Bradford 25138-9). *Editorial Communications* should be addressed to *The Editor*, at the same address.

### Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal* —

#### LECTURES

The Disperse Dyes — their Development and Application

*R. K. Fourness*

Finishing of Fabrics containing Terylene Staple Fibre

*N. M. Mims*

#### F.T.C.C. PUBLICATION

Early Work on Fixing Standards for Light Fastness

*Fastness Committee*

#### COMMUNICATIONS

Light Fastness Assessments of Dyed Textiles  
and their Bearing upon the Mechanism of Fading

*J. C. Eaton and C. H. Giles*

Photochemical Oxidation of Wool in the  
Presence of Fluorescent Compounds

*D. R. Graham and K. W. Statham*

The Desorption of Level-dyeing Acid Dyes from Wool

*C. H. Nicholls*

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## FORTHCOMING MEETINGS OF THE SOCIETY

### Friday, 21st September 1956

MANCHESTER SECTION. *Structural Aspect of Cellulosic Fibres*. Dr. N. S. Wooding (Courtaulds Ltd.) Textile Institute, 10 Blackfriars Street, Manchester. 7 p.m.

### Tuesday, 25th September 1956

SCOTTISH SECTION. *Man-made Fibres in the Carpet Trade—Dyeing of Staple and Yarns*. R. C. Cheetham, Esq., A.M.C.T., F.R.I.C. (Courtaulds Ltd.). St. Enoch Hotel, Glasgow. 7.15 p.m.

### Friday, 28th September 1956

NORTHERN IRELAND SECTION. Perkin Centenary Dinner and Ball. Woodbourne House Hotel, Suffolk, Dunmurry. Reception 7.30 p.m., Dinner 8 p.m.

SCOTTISH SECTION. Perkin Centenary Dinner-Dance—Ca'doro Restaurant, Glasgow. 7 p.m. for 7.30 p.m.

### Wednesday, 3rd October 1956

MIDLANDS SECTION. *A New Method for the Continuous Dyeing of Hydrophobic Fibres*. D. A. Garrett, Esq., M.A. King's Head Hotel, Loughborough. 7 p.m.

### Thursday, 4th October 1956

NORTHERN IRELAND SECTION. *Man-made Fibres with Special Reference to their behaviour alone and in Blend Form on Modified Flax Machinery*. R. G. Cairns, Esq. (Courtaulds Ltd.). Joint Meeting with Textile Institute, Kensington Hotel, Belfast. 7.30 p.m.

### Friday, 5th October 1956

LONDON SECTION. *Reactive Dyes for Textiles*. T. Vickerstaff, Esq., M.Sc., Ph.D., A.R.I.C., F.S.D.C. (I.C.I. Ltd.). Royal Society, Burlington House, London W.1. 6 p.m.

### Wednesday, 10th October 1956

NORTHERN IRELAND SECTION. *Fading Lamps—A Curse or a Blessing*. K. McLaren, Esq., B.Sc., F.R.I.C., F.S.D.C. Thompson's Restaurant, Donegal Place, Belfast. 7.30 p.m.

### Friday, 12th October 1956

WEST RIDING SECTION. Supper and Smoker, Victoria Hotel, Bradford. 7.30 p.m.

### Tuesday, 10th October 1956

SCOTTISH SECTION. *The Life and Work of Perkin*. Professor J. Read, Ph.D., M.A., F.R.S. St. Enoch Hotel, Glasgow. 7.15 p.m.

### Friday, 10th October 1956

LONDON SECTION. Fourth London Lecture. *Colour in Fashion*. James Laver, Esq., C.B.E. (Keeper of the Departments of Textiles and Engravings and Illustrations and Design, The Victoria and Albert Museum). Waldorf Hotel, London W.C.2. 7 p.m.

MANCHESTER SECTION. *A New Class of Dyestuffs—The Behaviour of Reactive Dyes in Dyeing and Printing*. T. Vickerstaff, Esq., M.Sc., Ph.D., A.R.I.C., F.S.D.C. (I.C.I. Ltd.). Textile Institute, 10 Blackfriars Street, Manchester. 7 p.m.

### Saturday, 20th October 1956

MIDLANDS SECTION. Perkin Celebration Dinner Dance, Welbeck Hotel, Nottingham. 7 p.m.

### Wednesday, 31st October 1956

MIDLANDS SECTION. *Reactive Dyes—A New Approach to Cellulose Dyeing*. T. Vickerstaff, Esq., M.Sc., Ph.D., A.R.I.C., F.S.D.C. Albert Hall Institute, Nottingham. 7 p.m.

WEST RIDING SECTION. Perkin Centenary Lecture—*The Life and Work of Perkin*. Professor John Read (University of St. Andrews). Hotel Metropole, Leeds. 7.30 p.m.

### Thursday, 1st November 1956

WEST RIDING SECTION. Perkin Centenary Lecture—*Early Stages in the Renaissance of the British Dye Industry. Tales from Turnbridge, Huddersfield, 1899-1920*. Dr. C. M. Whittaker, Esq., D.Sc., F.T.I., F.S.D.C. Victoria Hotel, Bradford. 7.30 p.m.

### Friday, 2nd November 1956

LONDON SECTION. *What the Dry Cleaner expects from Textiles and Textile Finishes*. D. D. McCordall, Esq., (Achille Serre) (Joint meeting with the London Centre of the Guild of Dyers and Cleaners). Royal Society, Burlington House, London W.1. 6.30 p.m.

WEST RIDING SECTION. Perkin Centenary Celebration Dinner and Dance. Victoria Hotel, Bradford. 7 p.m.

### Thursday, 8th November 1956

WEST RIDING SECTION. *A New Approach to Dyeing*. Lecturers, L. Peters, Esq., M.Sc., Ph.D., and C. B. Stevens, Esq., Ph.D., F.S.D.C. (The University of Leeds). Victoria Hotel, Bradford. 7.30 p.m.

### Tuesday, 13th November 1956

SCOTTISH SECTION. *Levelling Problems in the Dyeing of Vat Dyes*. H. Musshoff Esq. (Cassella). St. Enoch Hotel, Glasgow. 7.15 p.m.

### Wednesday, 14th November 1956

NORTHERN IRELAND SECTION. Perkin Centenary Lecture—*Life and Work of Perkin*. J. Boulton, Esq., M.Sc. (Tech.), F.R.I.C., F.T.I., F.S.D.C. (Courtaulds Ltd.). (This lecture will be followed by a buffet supper.) Thompson's Restaurant, Donegal Place, Belfast. 7.30 p.m.

### Thursday, 22nd November 1956

WEST RIDING SECTION. Lecture. Details later. Leeds. 7.30 p.m.

MIDLANDS SECTION. *Applications of Colour Physics to Textiles*. (A simple account for the practical man.) J. C. Guthrie Esq., M.Sc., F.Inst.P., F.T.I., and P. H. Oliver Esq., B.Sc., Grad.Inst.P. College of Technology, Leicester. 7 p.m.

MANCHESTER SECTION. Ladies' Evening. (Date and Time to be announced later.)

### Thursday, 6th December 1956

WEST RIDING SECTION. *Reactive Dyes—A New Approach to Cellulose Dyeing*. T. Vickerstaff, Esq., M.Sc., Ph.D., A.R.I.C., F.S.D.C. (I.C.I. Ltd., Dyestuffs Division). Victoria Hotel, Bradford. 7.30 p.m.

### Friday, 7th December 1956

LONDON SECTION. *The Chemical Revolution in Textile Technology*. Professor J. B. Speakman, D.Sc., F.R.I.C., F.T.I. (Leeds University). (Joint meeting with the London Section of the Textile Institute.) Royal Society, Burlington House, London, W.1. 6 p.m.

### Tuesday, 11th December 1956

SCOTTISH SECTION. *Full Shades on Secondary Acetate and 'Tricel' and Allied Topics*. H. C. Olpin, Esq., M.Sc., F.R.I.C., F.S.D.C. and J. Wood, Esq., B.Sc., A.R.I.C. (British Celanese Limited). St. Enoch Hotel, Glasgow. 7.15 p.m.

### Wednesday, 12th December 1956

NORTHERN IRELAND SECTION. *Printing of Nylon and other Polyamide Fibres*. R. E. Fletcher, Esq., B.Sc., A.T.I. (The Clayton Dyestuffs Co. Ltd.). Thompson's Restaurant, Donegal Place, Belfast. 7.30 p.m.

MIDLANDS SECTION. *The Finishing of Garments and Fabrics of Bulked Terylene*. G. McLeavy, Esq., B.Sc., Ph.D., A.R.I.C. King's Head Hotel, Loughborough. 7 p.m.

## FORTHCOMING MEETINGS OF THE SOCIETY—continued

### Friday, 14th December 1956

MANCHESTER SECTION. *Pigment Printing—The Possibilities and Limitations*. Dr. Kass (Fabriken Bayer, A.G.). Textile Institute, 10 Blackfriars Street, Manchester. 7 p.m.

### Wednesday, 9th January 1957

NORTHERN IRELAND SECTION. *Application of Colour Physics to Textiles*. P. H. Oliver, Esq., B.Sc., Grad.Inst.P. and J. C. Guthrie, Esq., M.Sc., F.Inst.P. (Courtaulds Ltd.). Thompson's Restaurant, Donegall Place, Belfast. 7.30 p.m.

### Thursday, 10th January 1957

MIDLANDS SECTION. *Developments in Analytical Methods in the Dyehouse Laboratory*. R. W. Richardson, Esq., Ph.D., B.Sc., A.R.I.C. and Z. Bergmann, Esq. (Joint with the Society of Chemical Industry). Gas Theatre, Nottingham. 7 p.m.

### Friday, 11th January 1957

LONDON SECTION. *Ladies' Evening. Kippers, Cocktails, Confectionery & Colour*. G. J. Chamberlin, Esq. (The Tintometer Ltd.). Royal Society, Burlington House, London, W.1. 6 p.m.

### Tuesday, 15th January 1957

SCOTTISH SECTION. *Application of Colour Physics to Textiles*. J. C. Guthrie, Esq., M.Sc., F.Inst.P., F.T.I. and P. H. Oliver, Esq., B.Sc., Grad.Inst.P. (Courtaulds Ltd.). St. Enoch Hotel, Glasgow. (Joint Meeting with Textile Institute.) 7.15 p.m.

### Friday, 18th January 1957

MANCHESTER SECTION. *A New Approach to the Dyeing of Hydrophobic Fibres*. D. Garrett, Esq. (I.C.I. Ltd.). Textile Institute, 10 Blackfriars Street, Manchester. 7 p.m.

### Wednesday, 23rd January 1957

MIDLANDS SECTION. *Problems in the Processing of Rayon Blends*. R. C. Cheetham, Esq., A.M.C.T., F.R.I.C., and H. D. Edwards, Esq., Ph.D., A.R.I.C. College of Technology, Leicester. 7 p.m.

### Friday, 1st February 1957

LONDON SECTION. *A Review of the Colour Fastness Requirements in Dyed Textiles*. T. H. Morton, Esq., M.Sc., Ph.D., F.S.D.C. (Courtaulds Ltd.). (Joint meeting with the London Section of the Clothing Institute.) Royal Society, Burlington House, London W.1. 6 p.m.

### Wednesday, 13th February 1957

NORTHERN IRELAND SECTION. *Fifty Years Dyeing Linen in Ireland*. N. McCraith, Esq. (Joint Meeting with Textile Institute.) Thompson's Restaurant, Donegall Place, Belfast. 7.30 p.m.

### Friday, 15th February 1957

MANCHESTER SECTION. *Problems in the Wet Processing of Rayon Blends*. H. D. Edwards, Esq., Ph.D., A.R.I.C. and R. C. Cheetham, Esq., A.M.C.T., F.R.I.C. (Courtaulds Ltd.). Textile Institute, 10 Blackfriars Street, Manchester. 7 p.m.

### Wednesday, 20th February 1957

MIDLANDS SECTION. *Basic Mechanisms of Winch Processing*. R. H. Waddington, Esq., B.Sc. Albert Hall Institute, Nottingham. 7 p.m.

### Friday, 8th March 1957

LONDON SECTION. *Soiling of Natural and Synthetic Fibres*. G. G. Taylor, Esq., B.Sc., A.Inst.P. (Clayton Dyestuffs Co. Ltd.). Royal Society, Burlington House, London W.1. 6 p.m.

### Wednesday, 13th March 1957

NORTHERN IRELAND SECTION. *Problems in Wet Processing of Rayon Blends*. H. D. Edwards, Esq., Ph.D., A.R.I.C. and R. C. Cheetham, Esq., A.M.C.T., F.R.I.C. (Courtaulds Ltd.). Thompson's Restaurant, Donegall Place, Belfast. 7.30 p.m.

### Wednesday, 20th March 1957

MIDLANDS SECTION. *Developments in Sizing as they affect the Dyer and Finisher*. J. H. MacGregor, Esq., Ph.D., F.R.I.C., and E. France, Esq., A.R.C.S., A.M.C.T. (Joint with the British Association of Chemists.) Midland Hotel, Derby. 7 p.m.

### Saturday, 23rd March 1957

NORTHERN IRELAND SECTION. Annual General Meeting and Dinner. Woodbourne House Hotel, Belfast. Details later.

### Thursday, 28th March 1957

MIDLANDS SECTION. *The Soiling of Natural and Synthetic Fibres*. G. G. Taylor, Esq., B.Sc., A.Inst.P. (Joint with the Textile Institute.) Carpet Trades Canteen, Kidderminster. 7.30 p.m.

### Friday, 29th March 1957

The Society's Annual Dinner. The Grosvenor House, Park Lane, London.

### Friday, 12th April 1957

MIDLANDS SECTION. Annual Dinner. George Hotel, Nottingham. 7 p.m.

### Wednesday, 17th April 1957

MIDLANDS SECTION. . . . and inwardly digest (a Dissertation on Technical Literature). S. Burgess, Esq., F.S.D.C., A.M.C.T. (Preceded at 6.30 p.m. by the Annual General Meeting of the Section). King's Head Hotel, Loughborough. 7 p.m.

### Friday, 3rd May 1957

MIDLANDS SECTION. The Mercer Lecture of the Society Grand Hotel, Leicester. 7 p.m.

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# THE JOURNAL

OF THE

## Society of Dyers and Colourists

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PUBLICATIONS SPONSORED BY THE SOCIETY'S FASTNESS TESTS  
CO-ORDINATING COMMITTEE—XIX

### Recommended Conditions for Fading Lamp Operation

LIGHT FASTNESS SUBCOMMITTEE

Many of the discrepancies between daylight exposures and those made in fading lamps arise from differences in the effective humidity during exposure. The average value occurring during daylight exposures in the United Kingdom is 20%, and fading lamps should be adjusted to operate at this effective humidity.

The Light Fastness Subcommittee of the Society of Dyers and Colourists has been investigating the problem of why many light fastness determinations made in fading lamps against the *B.S. 1006* standards<sup>1</sup> differ significantly from exposures made in daylight. A detailed discussion of the possible causes was published in the Society's *Journal* for December 1954<sup>2</sup>, when the view was expressed that the greatest discrepancies would occur when there are wide variations in the effective humidity of the air surrounding the pattern and standards during exposure.

The "effective humidity" is a complicated factor which takes into account, not only the relative humidity of the surrounding air, but also its temperature, the surface temperature of the pattern, and the intensity of the light; its importance in light fastness testing has now been confirmed, and details of this experimental work will be published in the *Journal* in the near future.

Briefly, it has been found that the effective humidity is always lower than the relative humidity of the surrounding air if the surface temperature is higher than the air temperature. Sunlight and the light from the enclosed carbon arc both cause surface heating, although in some lamps this is minimised by the provision of a heat filter in the form of a glass and water screen between the globe and the patterns. In daylight testing in Great Britain the average effective humidity is about 20%; exposures of humidity-sensitive patterns made in fading lamps operating at lower values will give results which are higher than daylight, whilst those made at higher values will give lower results. The magnitude of the difference naturally depends on the sensitivity of the pattern being tested and how far the effective humidity departs from the daylight value, and results as much as

two grades higher at an effective humidity of less than 5% and three grades lower at 80% have been observed.

The following notes indicate the possibility or otherwise of achieving the required effective humidity in each of the lamps which were considered by the Subcommittee during the period of the investigation. Those currently available which are capable of producing the desired conditions are indicated by an asterisk (\*).

ATLAS ELECTRIC DEVICES Co. (Chicago, U.S.A.)

*Model C*—marketed between 1919 and 1928

*Model CV*—marketed between 1928 and 1932

These lamps do not contain a water screen and are not humidified: the effective humidity is less than 5%.

*Model FDA*—marketed between 1932 and 1941

*Model FDA-R*—first marketed in 1941 and currently available

These lamps are humidified but not screened, and should be operated under the conditions recently specified by the makers<sup>3</sup>. These give the highest effective humidity possible with this type of lamp, but even so, the value is only 5–10%, as the surface temperature is about 60°F. higher than the air temperature. The effective humidity is therefore too low to duplicate British daylight conditions†.

CALICO PRINTERS ASSOCIATION LTD. (Manchester)

\**C.P.A. Fading Lamp*—first marketed in 1934 and currently available

This lamp is screened and humidified, and should be operated so that the air in the exposure

† This effective humidity is very close to that occurring in the AATCC Sunlight Test, which the lamp is intended to duplicate.

chamber has a *relative* humidity of  $30 \pm 10\%$ ; this can be measured with a wet-and-dry-bulb thermometer with the bulbs shielded from direct radiation from the arc. If this value cannot be readily achieved, the makers should be consulted.

KELVIN, BOTTOMLEY & BAIRD LTD. (Glasgow and London)

*Original Model*—marketed between 1924 and 1928

This lamp is unscreened and not humidified; the effective humidity is less than 5%.

*Fugitometer*—marketed between 1928 and 1942

This lamp carries a plate stating that it was developed in collaboration with the British Research Association for the Woollen and Worsted Industries (now the Wool Industries Research Association). It is humidified and unscreened, and should be operated with water in the tray beneath the table, which gives an effective humidity of about 20%.

*Fugitometer*—marketed between 1945 and 1953

This lamp is screened and humidified, and should be operated so that the air in the exposure chamber has a *relative* humidity of  $30 \pm 10\%$ ; this can be measured with a wet-and-dry-bulb thermometer with the bulbs shielded from direct radiation from the arc.

KELVIN & HUGHES (INDUSTRIAL) LTD. (Glasgow and London), successors to K.B.B. Ltd.

*\*Fugitometer*—first marketed in 1953 and currently available.

This lamp is screened and humidified, and should be operated so that the air in the exposure chamber has a *relative* humidity of  $30 \pm 10\%$ ; this can be measured with a wet-and-dry-bulb thermometer with the bulbs shielded from direct radiation from the arc. The desired conditions can usually be obtained by operating the lamp with the water heater and the humidifying jet turned off.

Whilst the preferred conditions for lamp operation will ensure that the results obtained are closest to daylight exposures made in Great Britain, the excess of ultraviolet radiation emitted by the enclosed carbon arc can cause anomalies in some cases, and this aspect has been discussed in a paper published in March 1956<sup>4</sup>. *Exposures in carbon-arc fading lamps, even under the preferred conditions of effective humidity, must not, therefore, be regarded as always equivalent to daylight testing, but rather as a means of obtaining a fairly reliable indication of the actual light fastness when time does not permit a daylight exposure to be made.*

The practice of expressing light fastness in terms of the number of hours of lamp exposure necessary to produce a certain amount of fading is completely unsatisfactory; assessments should always be made against the standards (B.S. 1006) exposed simultaneously.

(Received 12th June 1956)

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- <sup>1</sup> B.S. 1006:1955 (London and Manchester: British Standards Institution).
- <sup>2</sup> McLaren, K., J.S.D.C., **70**, 553 (1954).
- <sup>3</sup> Norton, J. E., *Amer Dyestuff Rep.*, **43**, P 628 (1954).
- <sup>4</sup> McLaren, K., J.S.D.C., **72**, 86 (March 1956).

### COMMUNICATIONS

## The Influence of Histological Factors on the Attack of the Wool Fibre by Various Reagents

R. L. ELLIOTT and J. B. ROBERTS

An investigation, including optical and electron microscope studies, of the sequence of attack of various reagents on wool has revealed significant variations in the stability of the histological components of the fibre. In particular, further information has been obtained about the resistant membranes, together with additional evidence of a tubular resistant membrane lying immediately beneath the cuticular layer, and completely surrounding the cortex. This membrane is identified with the subcuticular membrane of Alexander and Earland<sup>1</sup>. The lability of the orthocortex, and the greater stability of the paracortex, towards hydrolytic reagents, have also been confirmed. Observations on the action of oxidising agents, such as *N*-chlorourea and acid potassium permanganate, are also included. Details are given of a new rapid technique for obtaining fibre replicas for electron-microscopic examination.

### Introduction

The many investigations of the complex histological structure of the wool fibre are gradually clarifying the picture. The work now reported arises from that of Alexander and Earland<sup>1</sup>, who gave the name "subcuticular membrane" to the residue obtained after treatment of the wool fibre with peracetic acid followed by ammonium hydroxide. Manogue and Elliott<sup>2</sup>, by similar treatment, isolated a cuticular sheath with pronounced scale markings, showing small sacs similar to those found in the Allwörden reaction and thus

originally located in the outer part of the cuticular layer. These workers also treated Chlorozymed wool, devoid of scales, with the same reagents, obtaining fibrils, microfibrils, and occasionally very thin membranes associated with the fibrous material. Mercer<sup>3</sup> believed that the cuticle sheath is more resistant than the cortex material, but Alexander, Zahn, and Haselmann<sup>4</sup> classed the cuticle sheath as a contaminant, and identified the inner fibrous component as the subcuticular membrane.

Previously, Zahn and Haselmann<sup>5</sup> had concluded



that part of the cortex of wool is more insoluble in certain reagents than the rest of the cortex. This conclusion is supported by the observed fibrillar structure of the insoluble residue from descaled fibres<sup>2</sup>, and Mercer<sup>6</sup> termed this more insoluble part the "paracortex".

Manogue, Moss, and Elliott<sup>7</sup> investigated further the evidence for a subcuticular membrane, and isolated residues resistant to various treatments, particularly to 2 N. aqueous sodium hydroxide solution at 60°C. These residues contained very thin membranes, originally located at the boundaries of each cortical cell. Fraser and Rogers<sup>8</sup> refute the existence of a subcuticle membrane, and interpret the cuticular sheath as comprising epicuticle and exocuticle, covering the membranous flap, which in its turn lies upon a continuous fibrillar layer.

The present authors have continued the study of the effect on wool of various reagents, particularly boiling N. and 2 N. hydrochloric acid and boiling 0.05 N. aqueous caustic potash. Other treatments investigated are those with oxidising agents, such as (i) *N*-chlorourea<sup>9</sup>, in which it is found that, unlike other shrink-resist treatments<sup>10</sup>, no degradation of the cuticle takes place (Fig. 10 and 11), reaction being confined to the inner part of the fibre, as claimed by Earland<sup>9</sup>; and (ii) acid solutions of potassium permanganate<sup>11</sup>, showing that the cystine fraction of the wool fibre is oxidisable by this treatment is located solely in the cuticle (Fig. 12).

### Experimental

#### 1. ACTION OF HYDROCHLORIC ACID ON WOOL

Degreased 64s Merino wool fibres (2 g.) were boiled with 2 N. hydrochloric acid (100 ml.). Samples of the fibre residues, and of the acid hydrolysing liquor, were removed at definite intervals. The fibre residues were washed with distilled water, stained with Methylene Blue, and examined under the optical microscope.

#### Microscopical Observations of Fibre Residues

(a) AFTER 10 MIN. ACID TREATMENT—Some degradation of the fibre has taken place, but no specific histological component appears to have been attacked (Fig. 1).

(b) AFTER 20 MIN.—A large number of cortical cells have been released into the liquor, and it can be seen (Fig. 2) that these are derived from one half only of the cortex.

(c) AFTER 30 MIN.—The free cortical cells have been sufficiently hydrolysed to dissolve in the acid liquor, and cortical cell membranes are left containing what is presumably paracortex. These membranes are surrounded by a loosely adhering continuous layer of cuticle material, which in many cases is stripping away from the cortex remnants (Fig. 3).

(d) AFTER 45 MIN.—At this stage the most noticeable feature is the break-up of one-half of the cortical cell membranes into short lengths. Segmentation is confined entirely to the orthocortex part of the fibre. A loose membrane is visible, surrounding the cortex (Fig. 4) (cf. Fraser and Rogers<sup>8</sup>).

(e) AFTER 90 MIN.—Almost the whole of each fibre has now dissolved. Only the curved segments of the orthocortex membranes remain (Fig. 5).

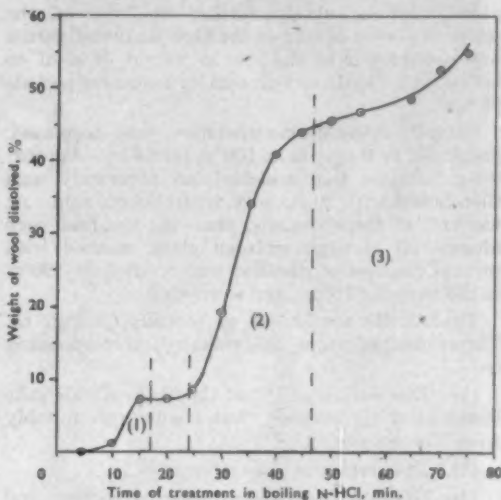


FIG. 15—Loss in Weight of Wool in Boiling N-HCl

This study of the action of acid on wool has revealed that attack is made first on the orthocortex (Fig. 2), leaving the paracortex and cortical membranes surrounded by a loosely adhering, continuous layer of cuticular material (Fig. 3), which differs from that previously isolated<sup>2</sup>, and is in no way fibrous in structure, as shown by its lack of birefringence under the polarising microscope. On more prolonged boiling the cortical cell membranes are found to segment in the orthocortex areas (Fig. 4), and finally only these membrane segments are left. Additional evidence of stepwise degradation has been obtained from loss-in-weight determinations on wool undergoing acid hydrolysis (Fig. 15).

#### Examination of Liquor Samples

The liquor samples were concentrated *in vacuo*, and then run on one-dimensional paper chromatograms to identify amino acid residues in the hydrolysates. The solvent used was the methyl ethyl ketone-water-propionic acid mixture of Clayton and Strong<sup>12</sup>. Amino acids were identified by running authentic samples of known amino acids at the side of each unknown mixture.

No free amino acids could be detected in the liquor samples after 10 min. acid treatment. In the sample taken at 20 min. the following amino acids were found— aspartic acid, glutamic acid, serine, threonine, and proline. The sample taken after 30 min. treatment contained all the normal amino acids of wool<sup>13</sup>.

The presence of aspartic and glutamic acids agrees with the findings of Blackburn<sup>14</sup>, and of serine and threonine with the known lability of peptide bonds involving the amino groups of these acids<sup>15</sup>. It is interesting to note that Zahn<sup>16</sup> has isolated peptides containing all these acids except

proline from aqueous extracts of wool made at room temperature.

#### *Loss in Weight of Wool during Acid Treatment*

In order to obtain further evidence of the selective attack of acid on the fibre, an investigation was carried out of the loss in weight of wool on boiling in *n*.<sup>\*</sup> hydrochloric acid for increasing periods of time.

Samples of 64s Merino wool fibres were degreased, and dried in the oven at 100°C. for 16 hr. Approx. 0.2-g. samples were weighed out accurately, and then boiled with 20 ml. of *n*. hydrochloric acid. At the end of the stipulated time the residues were filtered off through sintered glass, washed with several changes of distilled water, dried at 100°C. in the oven for 16 hr., and reweighed.

The results are shown graphically in Fig. 15. Three distinct steps are revealed, corresponding to—

- (1) Dissolution of 7% of the fibre, of indefinite histological significance, but coming presumably from the cuticle
- (2) Dissolution of the orthocortex
- (3) Gradual dissolution of the paracortex and resistant membranes.

### 2. ACTION OF ALKALIS ON WOOL

#### (a) *Aqueous Potassium Hydroxide*

Degreased 64s Merino wool fibres (2 g.) were boiled with 0.05 *N*. potassium hydroxide (100 ml.), and samples of the fibres were withdrawn at 10-min. intervals, washed carefully, stained with Methylene Blue, and examined under the optical microscope.

(i) **AFTER 10 MIN.**—Approximately one half of the cortex of the fibres has dissolved, leaving a tubular membrane. This membrane shows scale markings and contains a strip of cortex material which always occupies the inner half of the curved fibres, i.e. which corresponds to the paracortex (Fig. 6).

(ii) **AFTER 20, 30, 40, and 50 MIN.**—The fibres show much the same appearance as described under (i).

(iii) **AFTER 60 MIN.**—The paracortex itself dissolves, leaving the tubular membrane, which still bears scale markings, and surrounds what are presumably the alkali-insoluble cortical membranes<sup>7</sup> (Fig. 7).

This tubular membrane is thus yet another alkali-resistant membrane, differing histologically from that surrounding the actual cortical cells, but also showing a similar superior resistance to acid treatment compared with the remainder of the wool fibre. Thus these two membranes, although histologically distinct, so far show no difference in chemical behaviour.

#### (b) *Aqueous Ethylamine*

Wool fibres were prepared as before, and treated in 5% and 33% (wt./vol.) aqueous amine solutions at room temperature for 14 days.

(i) **5% AMINE**—The solution becomes cloudy, and there is no trace of the original wool structure. Microscopic examination shows that the scales, the subcuticular membrane, and the cortical cell membranes have all dissolved, leaving only cortical cells (Fig. 8). Such residues as there are stain heavily with Methylene Blue.

(ii) **33% AMINE**—The solution again becomes cloudy, leaving no trace of the original fibre structure, but microscopic examination shows that in this case the cloudiness is due not to cortical cells but to short lengths of membrane (Fig. 9), which are presumably cortical cell membrane. All scales and cortical cells have dissolved.

### 3. ACTION OF OXIDISING AGENTS ON WOOL

#### (a) *Acid Aqueous Solution of N-Chlorourea*

Previous workers<sup>10</sup> have studied the effect of shrink-resist processes on the cuticle of the wool fibre by electron-microscopic studies of fibre replicas. Earland<sup>9</sup>, in a study of the efficacy of organic *N*-chloro compounds as shrink-resist agents, has reported that solutions of *N*-chlorourea impart a degree of shrink resistance to wool, and he has postulated that the attack of this reagent is confined to reaction within the fibre, i.e. beneath the cuticular layer.

In the present work a sample of 64s Merino wool was treated, at a liquor ratio of 100 : 1, with 4% on its weight of available chlorine in the form of *N*-chlorourea, in 0.5 *N*. sulphuric acid containing 10% of sodium chloride, at 18°C. for 30 min. The treated wool was subsequently examined under both optical and electron microscopes (in the latter case a new rapid technique for preparation of fibre replicas was used—see Appendix).

It appears (Fig. 10 and 11) that no degradation of the cuticle has taken place, whereas in wet chlorination and in other processes a large part of the exocuticle is dissolved away, leaving the characteristic ridged structure of the endocuticle<sup>10</sup>.

#### (b) *Acid Potassium Permanganate Solutions*

Alexander, Fox, and Hudson<sup>11</sup> have reported that only 25% of the cystine content of wool is oxidisable by acid potassium permanganate solutions. In order to determine whether this fraction has any histological significance, solvent-extracted 64s Merino wool fibres (0.5 g.) were treated with a 2% solution of potassium permanganate in 0.5 *N*. sulphuric acid at 17°C. for 10 min. The wool became heavily pigmented at the sites of oxidation.

After being rinsed in distilled water, the fibres were examined under the optical microscope. The fibres appeared uniformly pigmented, with no difference between orthocortex and paracortex.

With the object of dissolving away the oxidised fraction of the fibre, the wool was treated on the microscope slide with a 10% aqueous caustic soda solution at room temperature. It was noticed that this treatment caused the cuticle to fragment. Pressure was then applied to the cover slip, whereupon sheets of cuticle material broke away from the fibre, revealing the completely unpigmented cortex (Fig. 12).

\* Changed from 2 *N*., as used in the microscopic investigation, in order that any steps in the graph might be more sharply defined.



FIG. 1—10 min.



FIG. 2—20 min.



FIG. 3—30 min.



FIG. 4—45 min.

FIG. 1-4—Wool boiled in 2N-HCl for Increasing Periods of Time (optical micrographs,  $\times 300$ )

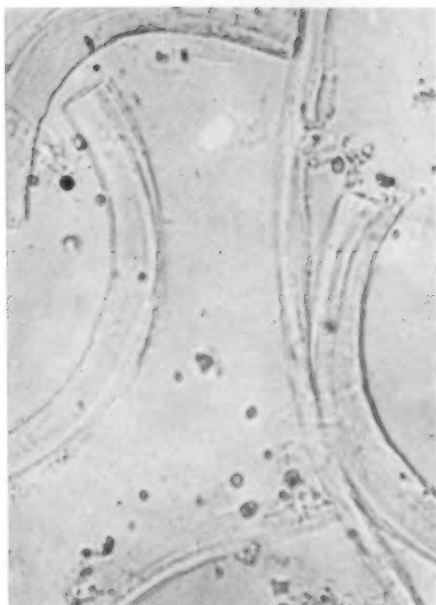


FIG. 5—Wool boiled for 90 min. in 2N-HCl (optical micrograph,  $\times 600$ )

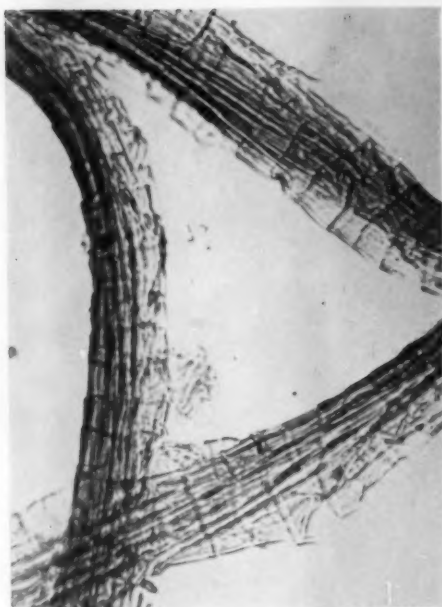


FIG. 6—Wool boiled for 10 min. in 0.05N-KOH (optical micrograph,  $\times 500$ )



FIG. 7—Wool boiled for 60 min. in 0.05N-KOH (optical micrograph,  $\times 500$ )



FIG. 8—Wool treated for 14 days in 5% Ethylamine Solution (optical micrograph,  $\times 500$ )





FIG. 9—Wool treated for 14 days in 33% Ethylamine Solution (optical micrograph,  $\times 500$ )



FIG. 10—*N*-Chlorourea-treated Wool (optical micrograph,  $\times 2000$ )



FIG. 11—Bedacryl 122X-Aluminum Replica of *N*-Chlorourea-treated Wool (electron micrograph,  $\times 5000$ )



FIG. 12—Wool oxidised with Acid  $\text{KMnO}_4$  and treated with 10%  $\text{NaOH}$  soln. (optical micrograph,  $\times 600$ )

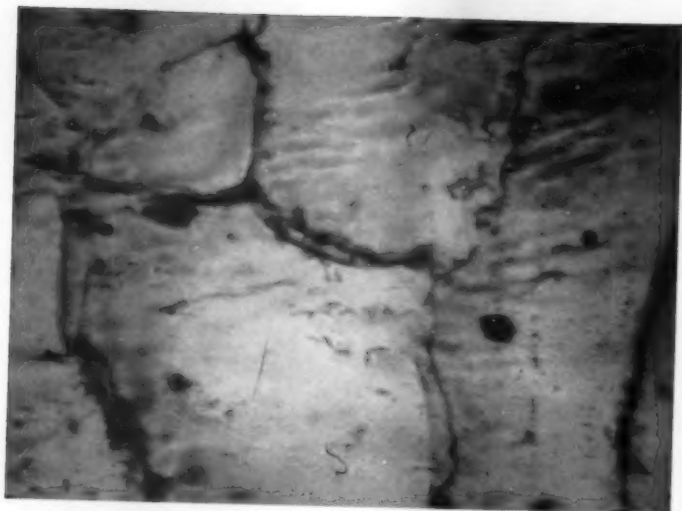


FIG. 13— Bedacryl 122X-Aluminium Replica of Chlorinated Wool  
(unshadowed electron micrograph,  $\times 5000$ )



FIG. 14— Bedacryl 122X-Aluminium Replica of Alkali-damaged Wool  
(silver-shadowed electron micrograph,  $\times 5000$ )

Precisely similar results were obtained after a period of oxidation treatment of 16 hr. It is therefore apparent that the cystine fraction which is oxidisable by acid permanganate is located solely in the cuticle. Since the cuticle forms only some 10% of the fibre, this implies that the cuticle contains a higher percentage of cystine than the remainder of the fibre, in accordance with the analyses of Geiger<sup>17</sup>.

### Appendix

#### A NEW TECHNIQUE FOR THE RAPID PREPARATION OF FIBRE REPLICAS FOR EXAMINATION UNDER THE ELECTRON MICROSCOPE

In place of the time-consuming polystyrene-silica<sup>18</sup> and polystyrene-aluminium replica techniques previously described<sup>2,10</sup>, the following method has been developed for use with wool and similar fibres.

A film of a readily acetone-soluble resin (Bedacryl 122X (ICI)<sup>19</sup>) is cast on glass by allowing a 10% solution in xylene to flow over a microscope slide, and dry without draining. About 50 wool (or other) fibres are then placed on the surface of the resin, and covered with another microscope slide. The sandwich of fibres is then heated on a hotplate at 80°C. for 10 min. under the pressure given by a 1-kg. weight.

After cooling, the fibres are mechanically stripped from the film, and the resulting replica is coated with aluminium by vacuum evaporation in the usual way. The combined resin-aluminium film is then divided into small squares (0.5 cm. side) by means of a razor blade. The squares are stripped mechanically from the slide, and mounted, aluminium side down, on grids supported on specimen holders. One drop of methylated spirits is placed on the resin and allowed to dry. This causes the replica to adhere to the grid. The cap is then placed on the specimen holder, and the whole is immersed in boiling 50:50 acetone-ether

to dissolve off the resin (5–10 min. is sufficient). After drying, the replica is ready for examination in the electron microscope. The total time of preparation is approx. 90 min. Two examples of this technique are shown in Fig. 13 and 14.

\* \* \*

The authors wish to thank Mr. W. Ingamells for his assistance in the development of the replica technique, and the Wool Textile Research Council for a scholarship (to J.B.R.).

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## A Study of the Photochemistry of Dyes on Proteins and other Substrates

J. W. CUMMING, C. H. GILES, and Miss A. E. McEachran

The rates of oxidation and of reduction in aqueous solution, and of fading in films of a typical protein (gelatin) and a typical non-protein (methylcellulose), have been determined for several series of dyes of different chemical classes. The general conclusion reached is that the photodegradation of a dye may follow either of the alternative mechanisms of oxidation or reduction, but normally oxidation occurs more readily on all non-proteins and reduction on proteins. In photochemical oxidation of dyes on non-proteins the substrate does not necessarily take part directly, but in reduction on proteins some constituent of the protein itself appears to act as the reducing agent. The histidine side-chains are probably the active constituent.

The conclusions in some instances have been confirmed by examination of spectral absorption curves of oxidation, reduction, and fading products of certain dyes.

Evidence has been given by many authors that fading on cellulosic fibres is due to oxidation\*, but no experimental work upon the nature of fading on protein fibres appears to have been reported, except the record of a previous investigation in this laboratory<sup>3</sup>. In this it was shown that

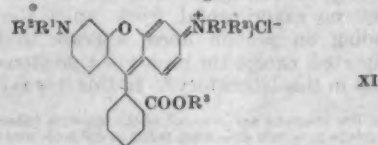
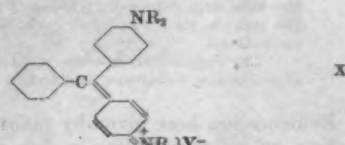
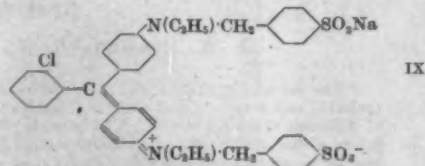
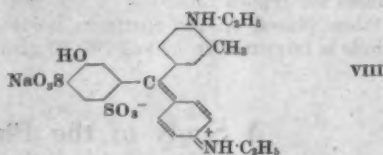
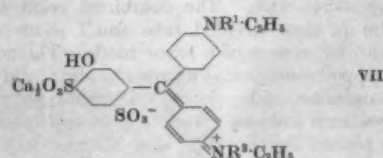
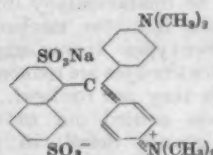
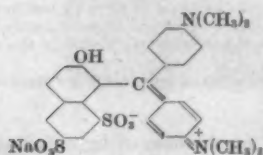
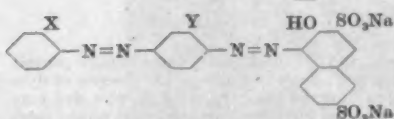
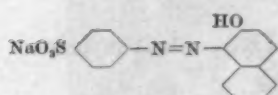
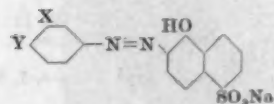
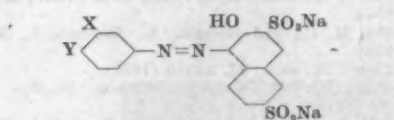
the chemical mechanism of light fading of certain azo dyes must be fundamentally different on all protein substrates from the mechanism on substrates of all other types. It was suggested that in fading on proteins the dyes are reduced, whereas on other substrates they are oxidised. The present investigation was carried out to examine this hypothesis in greater detail; in particular to

\* A few examples are on record of the apparent reduction of specific groups in certain dyes when faded on cellulosic substrates *cf.* 2.

TABLE I

## Selection of Dyes Investigated

| Dye   | C.I. No. | Formula |
|---|----------|---------|
| ACID DYES                                       |          |         |
| <i>Sulphonated Hydroxyazo Dyes</i>              |          |         |
| Orange R (phenylazo-R acid) ... ..              | 28       | I       |
| — and derivatives ... ..                        | —        | I       |
| Cochineal Scarlet (phenylazo-oxy-L acid) ... .. | 25       | II      |
| — and derivatives ... ..                        | —        | II      |
| Orange II ... ..                                | 151      | III     |
| Cloth Red 2R (NAC) ... ..                       | 253      | IV      |
| Dimethyl derivative ... ..                      | —        | IV      |
| <i>Sulphonated Diphenylnaphthylmethanes</i>     |          |         |
| Lissamine Green BN (ICI) ... ..                 | 737      | V       |
| Lissamine Green V (ICI) ... ..                  | 735      | VI      |
| <i>Sulphonated Triphenylmethanes</i>            |          |         |
| Disulphine Blue A (ICI) ... ..                  | 714      | VII     |
| Disulphine Blue V (ICI) ... ..                  | 712      | VII     |
| Xylene Cyanole FF (S) ... ..                    | 715      | VIII    |
| Disulphine Green B (ICI) ... ..                 | 667      | IX      |
| BASIC DYES                                      |          |         |
| <i>Triphenylmethanes</i>                        |          |         |
| Brilliant Green Y ... ..                        | 662      | X       |
| Malachite Green OS ... ..                       | 657      | X       |
| <i>Xanthen Dyes</i>                             |          |         |
| Rhodamine B ... ..                              | 749      | XI      |
| Rhodamine 6GB (ICI) ... ..                      | —        | XI      |





discover—(a) whether the difference in behaviour of proteins and other substrates in dye fading applies to other types of dyes than those previously used, (b) whether the difference can be ascribed to reduction on proteins, and, if so, (c) whether the constituent of the protein molecule which is responsible can be identified. The method used was to compare the rates of oxidation, reduction, and fading for series of various types of dye, and also to examine the absorption spectra of the products of these reactions.

## Results and Discussion

### GENERAL METHODS

At the outset it seemed likely that three general methods might be used to detect oxidation or reduction during irradiation—

(a) Measurement of the relative rates of oxidation and reduction of various series of dyes in solution, and of their fading on typical substrates of the two classes, protein and non-protein, the different dyes of a given series having the same fundamental structure but containing small substituent groups of differing degrees of electro-negativity situated in the *meta* or the *para* position to the chromophoric centre, or in other orientations.

The relative rates of oxidation and of reduction of dyes of any such series should, of course, vary in the opposite direction if the reaction site is the

same in each case, because ease of oxidation requires at the reaction site a high electron density, and ease of reduction a low electron density. The data are conveniently plotted as ordinates against the Hammett  $\sigma$ -values of the respective groups\* as abscissae *cf.* 3.

(b) Comparison of light-absorption spectra of irradiated dyes with those of their oxidation and reduction products.

(c) Examination by chromatography of degradation products from exposed dyed material. (Couper<sup>5</sup> has used this method for dyes on cellulose acetate.)

All three methods have now been examined, but the complex nature of the mixtures of degradation products obtained makes the results of (b) and (c) rather inconclusive, and method (a) appears to be more reliable.

### SELECTION OF DYES

The dyes used were the most suitable available and comprised the series shown in Table I.

Anthraquinone dyes were not used because of their low rates of fading.

\* The  $\sigma$ -value of a group is the logarithm of the ratio of the ionisation constant of benzoic acid substituted with the group to that of unsubstituted benzoic acid. It is thus a measure of the electron-attracting power of the group. A group with a high  $\sigma$ -value, e.g. the nitro group, will tend to increase ionisation of another substituent *meta* or *para* to it in a benzene nucleus. Thus *p*-nitrophenol is a stronger acid than phenol itself.

TABLE II  
Relative Rates of Degradation of Pairs of Acid and Basic Dyes

( $t_F$  = time for 10% loss of dye)

| Dye                | Oxidation<br>$t_F$<br>(sec.) | Reduction<br>$t_F$<br>(sec.) | Cellofas A                    |                | $\lambda$<br>( $\text{\AA}$ ) | Fading*<br>Gelatin<br>Cast<br>$t_F$<br>(hr.) | Dyed<br>$t_F$<br>(hr.) | Cellofas A<br>+ Histidine     |                |
|--------------------|------------------------------|------------------------------|-------------------------------|----------------|-------------------------------|--|------------------------|-------------------------------|----------------|
|                    |                              |                              | $\lambda$<br>( $\text{\AA}$ ) | $t_F$<br>(hr.) |                               |  |                        | $\lambda$<br>( $\text{\AA}$ ) | $t_F$<br>(hr.) |
| Cloth Red 2R (IV)  | 3(a)†                        | 5(b)†                        |                               |                | 5200                          | 27.5   |                        |                               |                |
| Dimethyl-IV        | 9(a)                         | 3(b)                         |                               |                | 5300                          | 3.0  |                        |                               |                |
| Lissamine Green BN | 9(a)                         | 5(c)                         | 6400                          | 0.1, 7.5       | 6400                          | 2.1, 7.5                                     |                        | 6400                          | 1.4, 7.5       |
| Lissamine Green V  | 16(a)                        | ‡(c)                         | 6500                          | 0.2, 31.0      | 6500                          | 2.0, 6.5                                     |                        | 6500                          | 0.6, 4.5       |
| Disulphine Blue V  | 18(a)                        | ‡(c)                         | 6400                          | 5.5, 45.0      | 6400                          | 2.0, 7.0                                     | —, 5.5                 |                               | 1.0, 7.0       |
| Xylene Cyanole FF  | 11(a)                        | 48(c)                        | 6200                          | 0.3, —         | 6200                          | 3.2, —                                       |                        |                               | 1.3, —         |
| Disulphine Blue A  | 36(a)                        | 660(d)                       | 6400                          | —, 37.5        | 6400                          | —, 6.5                                       | —, 3.5                 |                               | —, 6.0         |
| Disulphine Green B | 64(a)                        | 240(d)                       | 6500                          | —, 12.0        | 6500                          | —, 2.5                                       |                        |                               |                |
| Brilliant Green Y  | 210(e)                       | 85(g)                        | 6300                          | 5.8, 22.0      | 6300                          | 2.5, 4.0                                     |                        |                               | 7.0, —         |
| Malachite Green OS | 70(f)                        |                              |                               |                |                               |  |                        |                               |                |
|                    | 85(e)                        | 220(g)                       | 6300                          | 0.5, 13.0      | 6300                          | 2.8, 7.0                                     |                        |                               | 13.5, —        |
|                    | 50(f)                        |                              |                               |                |                               |  |                        |                               |                |
| Rhodamine B        | 30(h)                        | 25(g)                        | 5500                          | 0.2, 14.0      | 5600                          | 0.3, 5.0                                     |                        |                               | 0.2, —         |
| Rhodamine 6GB      | 60(h)                        | 250(g)                       | 5300                          | 1.0, 36.0      | 5400                          | 4.2, 8.0                                     |                        |                               | 0.8, —         |

\* The first and second figures in each case refer to the initial fade and the subsequent slow fade respectively.

† Details of oxidation and reduction tests—

0.5 c.c. dye solution (0.0005 M.) + reagent, aqueous soln., at room temp.

Reagents—

(a) 8.5 c.c. of 10%  $K_2S_2O_8$

(b) 8.5 c.c. of 0.5%  $Na_2S_2O_4$

(c) 8.5 c.c. of 5%  $Na_2S_2O_4$

(d) 8.5 c.c. of 2.5%  $TiCl_3$

(e) 20 c.c. of 20-vol.  $H_2O_2$

(f) 10 c.c. of 0.5%  $K_2S_2O_8$  in pH 6.0 buffer

(g) 14.5 c.c. of 0.005%  $Na_2S_2O_4$

(h) 10.5 c.c. of 2.2%  $NaClO$

‡ Decolorisation too rapid to measure.

TABLE III  
Position of Peaks (A.) in Spectra of Degradation Products

| Dye                          | Oxidation<br>in Soln.                      | Reduction<br>in Soln.                         | Cellofas A                 | Fading in<br>Gelatin                                   | Cellofas A +<br>Histidine                                |
|------------------------------|--|---|----------------------------|--|--|
| <i>p</i> -Phenetidine→R acid | (2750)*<br>(3500)<br>(3700)<br>(4000)      | (2900)*<br>(3300)<br>(3550)<br>3600<br>(3700) | 2900<br>(3100)†<br>(3300)  | (2600)*<br>(2900)<br>(3300)                            |  |
| Orange II                    | 3300                                       | (2600)<br>3000<br>(3100)<br>3400<br>3700      | (2600)†                    | (2600)<br>2900<br>(3400)<br>(3700)<br>(4000)<br>(4500) |  |
| Disulphine Blue A            | (2700)<br>3600<br>4300                     | 3100<br>3400<br>(4000)                        | (2400)†<br>(3500)          | (2700)<br>(3400)<br>(3600)<br>(4000)<br>(4500)         |  |
| Disulphine Blue V            | (2900)<br>(3350)<br>3700<br>(3800)<br>4300 | 3100<br>(>4000)                               | (3800)<br>(4100)<br>(4300) | 2700<br>(3300)<br>3600<br>4000<br>(4500)               |  |
| Disulphine Green B           | (3100)<br>3600<br>(4200)                   | 3000<br>(3400)<br>(3700)                      | (2500)†                    | (2900)<br>(3400)<br>(3700)<br>(4800)                   | (2600)<br>(3000)<br>(3400)<br>(3500)<br>(3700)<br>(4500) |
| Malachite Green A            | (3000)<br>(3300)<br>3650                   | 2600<br>(2900)<br>3400<br>3700                | 2400†                      | 2900<br>(3400)<br>3700<br>4300                         | 3300<br>(3700)   |

\* Figures in parentheses indicate the estimated positions of indefinite peaks.

† Obtained by removal and water extraction of faded film (see Experimental Section). Fades on silica support gave no definite peaks.

#### SELECTION OF SUBSTRATES

It has already been shown that a variety of non-proteins appear to behave similarly in the fading of certain azo dyes, and several proteins also behave similarly to each other and differently from non-proteins<sup>5</sup>. The choice of substrates for use in this work could thus be made from a wide selection of materials, but water-soluble products which could easily be mixed with dye solutions and cast into clear transparent films were preferred. Cellofas A (ICI) (methylethylcellulose) and gelatin were therefore chosen as typical non-protein and protein materials.

The results are given in Tables II and III and in Fig. 1-4.

#### RELATIONSHIP BETWEEN RATES OF OXIDATION, REDUCTION, AND FADING

In all the series of azo dyes (Fig. 1-4) the sequence of oxidation rates† is the reverse of that of reduction rates, in accordance with prediction. The orders of fading rates on Cellofas A and gelatin

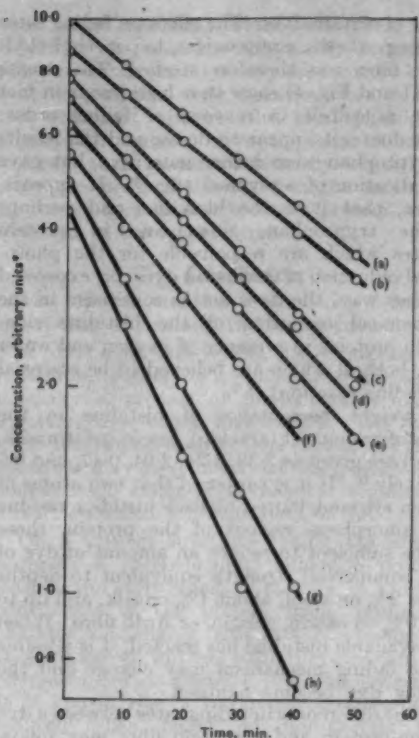
are parallel to those of oxidation and reduction respectively.

Comparisons made by selecting any pair of dyes in Table II, except the Rhodamines, show that the relative order of oxidation rates with the same oxidant is always the reverse of that of the reduction rates with the same reducing agent. This applies whether the dyes compared are very similar in structure, e.g., Brilliant Green and Malachite Green, or not, e.g., Disulphine Green B and Lissamine Green V. This is in accordance with prediction. The anomaly of the Rhodamines must be attributed to their reaction sites for oxidation and for reduction being different.

There are also corresponding parallelisms in fading rates. Thus, when comparisons are made within the pairs of dyes which are bracketed together in Table II, i.e. those most closely similar in structure, the order of fading rates on Cellofas A parallels the oxidation rates and the order of fading rates on gelatin parallels the reduction rates. This applies to the Rhodamines also, where all the four rates are higher for the B brand.

When comparisons of fading rate are made within pairs of dyes which differ more in structure

† Badger and Lewis<sup>7</sup> also obtained a linear relationship with negative slope, for the oxidation (with perbenzoic acid) of a series of substituted azobenzene derivatives.



| Curve | Substituent                | $\sigma$ -Value |
|-------|----------------------------|-----------------|
| (a)   | $p\text{-O-CH}_3$          | -0.27           |
| (b)   | $p\text{-O-C}_2\text{H}_5$ | -0.25           |
| (c)   | $p\text{-CH}_3$            | -0.17           |
| (d)   | —                          | 0               |
| (e)   | $p\text{-Cl}$              | +0.23           |
| (f)   | $m\text{-Cl}$              | +0.37           |
| (g)   | $m\text{-NO}_2$            | +0.71           |
| (h)   | $p\text{-NO}_2$            | +0.78           |

FIG. 1—Rates of Reduction of Aniline-R-acid Dyes (with Stannous Chloride)

\* The curves have been separated on the y-axis, for clarity.

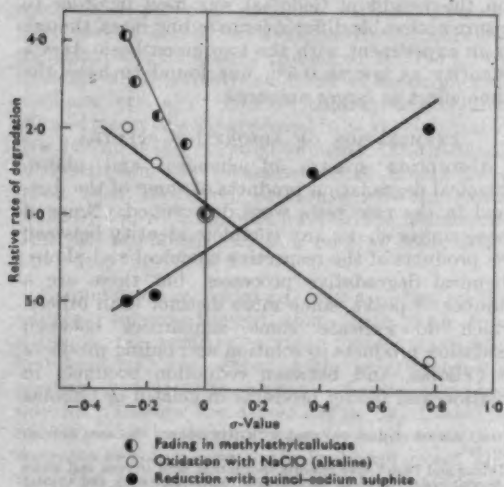


FIG. 3—Relation between Relative Rates of Fading, Oxidation, and Reduction, and  $\sigma$ -Value for Phenylazo-oxy-L-acid Dyes

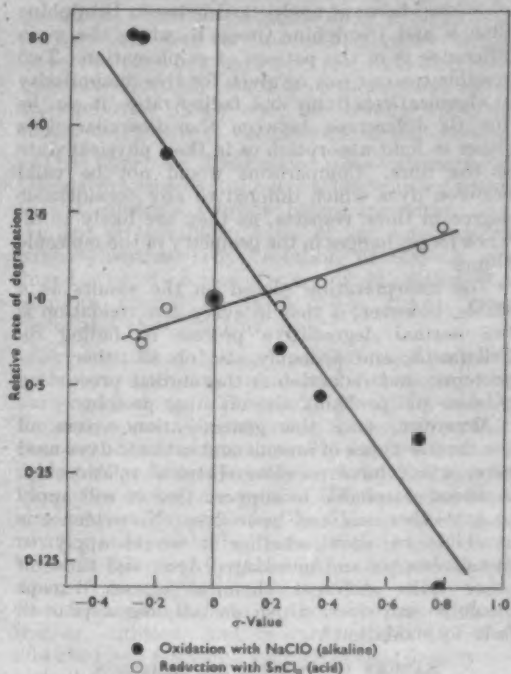


FIG. 2—Relation between Relative Rates of Oxidation and Reduction, and  $\sigma$ -Value for Phenylazo-R-acid Dyes

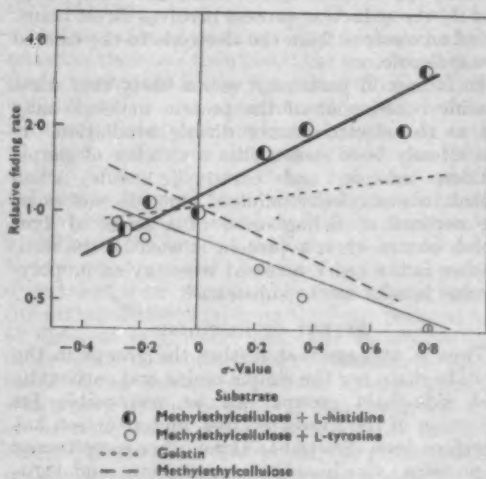


FIG. 4—Relation between Relative Fading Rate and  $\sigma$ -Value of Phenylazo-R-acid Dyes

than those bracketed, the parallelisms with oxidation and reduction are not observed. This applies (see Table II) to comparisons of Disulphine Blue V with Disulphine Blue A, where the A brand has two additional benzene nuclei; and between Disulphine Blue V and Disulphine Green B, where the main difference is in the pattern of sulphonation. Two possible reasons can be given for this dissimilarity in chemical reactivity and fading rates: it can be due to differences between the dissimilar dyes either in light absorption or in their physical state in the fibre. Comparisons would not be valid between dyes which differed to any considerable degree in these respects, as they are likely to do when large changes in the geometry of the molecule occur.

The interpretation placed on the results as a whole, however, is that in every case oxidation is the normal degradative process in fading on Cellofas A, and probably also on all other non-proteins; and reduction is the normal process on gelatin, and probably also on other proteins.

Moreover, since this generalisation covers all the diverse types of anionic and cationic dyes used here, which have no close chemical relationship, it seems reasonable to suppose that it will apply to any other acid and basic dyes. No evidence is available to show whether it would apply to metal-complex and mordanted dyes, and those of other quite different chemical classes, except insoluble azo dyes, which on cellulose appear to fade by oxidation<sup>3</sup>.

#### NATURE OF THE FADING REACTION

Hillson and Rideal<sup>11</sup> have studied the mechanism of photodegradation of some azo and triphenyl-methane dyes, using the Bequerel effect (production of a photocurrent by illuminating a platinum electrode coated with dye). They showed that either oxidation or reduction of dye could occur, according to the experimental conditions, and suggested that (a) the oxidation process is one of addition to the dye of hydroxyl radicals produced by reaction of excited dye molecules with water, and (b) the reduction process involves direct transfer of an electron from the electrode to the excited dye molecule.

In fading on proteins it seems likely that some specific constituent of the protein molecule may act as the electron donor during irradiation. It has already been shown that a number of simple amides, amines, and carboxylic acids, when added to methylethylcellulose films, do not cause the reversal of fading order of a series of dyes which occurs when a protein is used as the film; neither is the order reversed when nylon or polyglycine is used as the substrate<sup>3</sup>.

#### EFFECT OF HISTIDINE

Thus it appears that neither the groups in the peptide chain nor the simple amine and carboxylic acid side-chain groups can be responsible for reduction of the irradiated dye, and attention has therefore been directed to three other constituents of proteins, viz. histidine, tryptophan, and tyrosine. These amino acids were found by Carter<sup>12</sup> to be photochemically oxidised in solution in

presence of certain dyes. The effect on fading rates of adding these compounds to methylethylcellulose films was therefore studied. The results (Table II and Fig. 4) show that histidine is in fact effective in causing a reversal of fading order; tyrosine does not appear to do so; and the results with tryptophan were rather indecisive, but gave some indication of a reversal effect<sup>3</sup>. It appears, therefore, that it is the histidine, and perhaps also the tryptophan, side-chains in protein substrates which are responsible for the photochemical reduction of irradiated dyes; or, expressed in another way, the dyes act as sensitizers in the photochemical oxidation of the histidine side-chains in proteins in presence of oxygen and water vapour, both of which are believed to be essential to the fading reaction<sup>13</sup>.

The weight percentages of histidine in the proteins of groundnut (arachin), casein, gelatin, silk, and wool are given as 2.33, 3.24, 2.94, 0.47, and 0.7 respectively<sup>13</sup>. If it is supposed that two atoms of hydrogen are contributed by each histidine residue in the amorphous regions of the protein, these would be sufficient to reduce an amount of dye of normal commercial strength equivalent to depths of about 2% on wool, about 1% on silk, and up to about 10% on casein, gelatin, or Ardil fibre. When all the available histidine has reacted, it is possible that the fading mechanism may change and the remaining dye become oxidised.

Thus the differences in fading rates between a dye on a non-protein and a protein fibre may follow the suggested pattern when the protein fibre is, say, casein or Ardil, for any depth of dyeing, but when it is silk or wool the relationship may perhaps apply only to weaker dyeings and to the earlier stages of fading of heavier dyeings. In the earlier investigation<sup>3</sup> there was, however, no evidence of any difference in the nature of fading of normal depths on gelatin, silk, or wool. Nothing is known of the effect of chemical modification of wool on dye fading.

In the present experiments up to 5% of histidine (on the weight of Cellofas) was used in order to ensure noticeable differences in fading rates, though in an experiment with the two green basic dyes a quantity as low as 0.5% was found to have the same effect as larger amounts.

#### EXAMINATION OF ABSORPTION SPECTRA

Absorption spectra of chemical and photochemical degradation products of some of the dyes used in the rate tests were determined. None of these curves shows any complete identity between the products of the respective chemical and photochemical degradative processes, but there are a number of peaks, some more distinct than others, which do indicate some similarities between oxidation products in solution and fading products in Cellofas, and between reduction products in solution and fading products in gelatin or Cellofas

\* Only one set of dyes was used with tryptophan. See also Note on p. 381.

† Dilung and Dain<sup>4</sup> have recently shown that both oxygen and water vapour are involved in the fading of Malachite Green and Crystal Violet (spread on glass as dried films from pure alcoholic solutions). Fading appeared to be caused by photochemical reaction of oxygen with the dye-water complex.



with added histidine. Details are given in Table III. The shape of the supposedly corresponding pairs of curves would in any case not be expected to be exactly the same, because, even though the two processes, viz. chemical and photochemical degradation, might follow the same course, it is unlikely that at any arbitrarily chosen period after the start of the reaction the ratios of the successive degradation products present would be the same in the two systems.

### Conclusion

The following tentative conclusion follows from the results of this and the earlier investigation.

A dye may fade either by oxidation or by reduction. Fading on any non-protein substrate is normally a process of photochemical oxidation involving dye, water, and oxygen. The substrate itself does not necessarily take part in this reaction, though it may do so in special cases, and it may, of course, itself suffer photochemical degradation in a reaction which is independent of the dye fading.

On a protein substrate fading appears to be normally a photochemical reduction process (at least in the early stages of fading), in which some constituent of the protein itself, probably the histidine side-chains, acts as the reducing agent. It is not known whether oxidation occurs in special circumstances on proteins, but this seems likely. It is known that a few particular dyes may be reduced when faded on non-protein fibres\*, and this leads to the general conclusion that reduction and oxidation are alternative photochemical mechanisms of fading on any substrate, but usually reduction occurs on proteins, and oxidation on non-proteins.

### Experimental

#### MATERIALS AND GENERAL PROCEDURE

The films were prepared from methylethylcellulose (Cellofas A (ICI)) and from pure photographic gelatin. The azo dyes were prepared and purified in the laboratory; the other dyes were commercial products supplied without diluents; they were checked for homogeneity by chromatography and for purity (found ca.  $95 \pm 3\%$ ) by titanous chloride analysis. Some of the triphenylmethane, etc. acid dyes on the alumina column showed traces of what appeared to be unsulphonated dye, but no major coloured impurity. The methods of irradiation and measurement of fading rates have been described previously<sup>2,3</sup>. An Osira (G.E.C.) 400-w. lamp was used as the illuminant. This has prominent emission lines at 3650, 4047, 4360, 5461, and 5780 Å.

Examination of the oxy-L acid azo dyes on the alumina column showed that those with substituents of negative  $\sigma$ -value were practically homogeneous, but those with substituents of positive  $\sigma$ -value were mixtures of *ortho* and *para* isomers. Though the 4-position in oxy-L acid is unoccupied, the sulpho group in the 5-position restrains coupling *para* to the hydroxyl group, and thus the azo group normally enters at the 2-

position. An attempt was made to purify these mixtures by collecting and evaporating the appropriate bands of solution from the columns, but the products appeared to contain substantial quantities of colloidal alumina and the percentage purity values were very low (of the order of 30%). Nevertheless, a few oxidation and reduction tests were attempted on solutions of these made up on an equimolar basis, and the results appeared consistent in spite of the presence of the alumina.

The films for irradiation were prepared as already described<sup>2,3</sup>, the dye solution being added to the substrate solution before spreading. For the non-protein films, 4 c.c. of azo (or 2 c.c. of triphenylmethane) dye solution (0.0005 M.) was mixed with 8 c.c. (or 10 c.c.) of 4% Cellofas A solution, poured on to a 2.5 in.  $\times$  4 in. glass plate, and then dried by heat.

For one pair of dyes, gelatin films were prepared both by casting as usual and by dyeing the hardened material. The results (Table II) were in the same sense in the two cases. This confirms that the rate differences are not merely due to differences in crystalline form of any dye crystals which may be formed on cooling the gelatin. The hardened films were prepared by spreading the films as usual, then treating them with 10% formaldehyde solution in the cold, following by thorough rinsing and drying again by heat, by the normal process. They were then immersed at 60°C. in dye solutions without further addition, and removed when the dye adsorbed had reached a suitable optical density for test (usually in about 30 min.). The films for spectral measurements after irradiation were cast directly on to transparent silica plates, of the correct size for insertion in the spectrophotometer, and dried in air at room temperature, to avoid cracking.

The histidine-, tryptophan-, and tyrosine-containing films were normally prepared from Cellofas A solutions containing 5.0% of the pure (L-) reagent, calculated on the weight of methylethylcellulose.

Fading rates on each series of dyes were determined at the same time under the same conditions. Some variations in the  $t_F$  values (Table II) for individual dyes are sometimes observed between films prepared at different times, presumably owing to slight differences in atmospheric conditions during setting of the films, but the relative differences between dyes do not alter in sense. Most dyes show an initial rapid fade, attributable to fading of the smallest particles present, followed by a much slower one. Values of  $t_F$  for any pair of dyes taken from the initial portion of the curves (by extrapolation) and from the later portions are of different magnitude, but their ratio does not differ much. The initial part of the curve sometimes shows inconsistencies, as already noted<sup>2</sup>, which may be due to physical changes in the film or in some cases perhaps to *cis-trans* isomeric changes in azo dyes. The initial dye concentration on which the  $t_F$  values are calculated is in the present cases that at the point where the measurements commence.

\* The reducing agent here must be either the fibre, e.g. nylon, or a portion of the dye itself.

## MEASUREMENT OF RATES OF OXIDATION AND REDUCTION

The measurements of rates of oxidation and reduction were made on a photoelectric (E.E.L.) test-tube absorptiometer. The dye and the reagent solution, chosen for its suitable reaction rate, were mixed in a stoppered tube, and the optical density was measured at intervals at room temperature. The choice of reagents and conditions is rather severely limited by the necessity of using dye solutions of suitable strength for direct colorimetric measurement and reagents which react therewith at a measurable speed. In a number of cases the reaction was not measurable: it occurred either instantaneously or virtually not at all, and in order to obtain a complete series of tests a

TABLE IV

## Light Absorption (%) of Rhodamine Films of Equivalent Concentration

|                   | 4047 Å.    |         | 5461 Å.    |         | 5780 Å.    |         |
|-------------------|------------|---------|------------|---------|------------|---------|
|                   | Cellofas A | Gelatin | Cellofas A | Gelatin | Cellofas A | Gelatin |
| Rhodamine B ...   | 1.0        | 1.5     | 91.8       | 91.3    | 58.8       | 59.5    |
| Rhodamine 6GB ... | 4.0        | <1      | 80.0       | 85.7    | <1         | <1      |

variety of reagents at a number of different concentrations were used. The rates for each pair (or series) of dyes, however, were measured with each chosen reagent under identical conditions at the same time.

## MEASUREMENT OF ABSORPTION SPECTRA OF PRODUCTS OF FADING

One series of these curves was determined on films cast on silica plates, as already described, but an earlier series was obtained by removing the dyed and faded films from the glass supports, since glass does not transmit short-wave ultra-violet radiation. Large dyed films (6½ in. × 4½ in. plates) were prepared and cut in half, one half being retained for reference; the other was irradiated, and after irradiation to about 50% fade was dissolved off in 75–100 c.c. of boiling distilled water, the solution being examined in a silica cell. The irradiated Cellofas A films dissolved extremely slowly, but the solution was removed for test when sufficient of the products was present for evaluation. Malachite Green appeared to be held especially tenaciously by this film. Spectra of oxidation or reduction products were measured in silica cells from the appropriate solutions, after buffering to pH 7. In order to ensure that the degradation had not proceeded too far, the spectra of oxidised, reduced, or faded dyes were measured when some unchanged dye still remained; its amount was determined from the optical density at the absorption peak, and a solution of fresh dye of this same strength was used in the blank cell. Thus when the absorption curve is plotted, the superimposed curve of unchanged dye is eliminated, and the result shows the curve for the degradation products alone.

All measurements of fading rates and absorption spectra were made on a Unicam SP 500 photoelectric spectrophotometer.

## Appendix

## EFFECTS OF DIFFERENCES IN SPECTRAL ABSORPTION BETWEEN DYES

In the discussion above no account has been taken of the possible effect of differences in light absorption between pairs of dyes in affecting their relative fading rates.

In all cases except the Rhodamines, which are discussed below, the observed changes seem most unlikely to be attributable to spectral effects, because (a) the light absorption characteristics of the dyes compared are very close (see Table II), and (b) any differences in fading rates produced in this way would in any case not be expected to involve the reversal of relative rates observed between the two types of film.

The Rhodamines, however, do not show the reversal effect, and moreover the relative rates are in the same sense as the respective light absorptions of these dyes at the principal emission wavelengths of the lamp near the dye absorption peaks, viz. at 5461 Å. and 5780 Å. (see Table IV). There was some ground, therefore, for believing that at least some part of the observed differences here might be due to differences in light absorption rather than in chemical reactivity. A test was made to check this point, in which these dyes were faded behind filters passing principally the 5461 Å. line, and also filters passing principally the 4047 Å. line, where the light absorption of the dyes is lower, with the results shown in Table V.

TABLE V

## Percentage Fade of Equivalent Concentrations of Rhodamine Dyes

| Dye and Exposure         | Waveband covering Emission Line |                         |
|--------------------------|---------------------------------|-------------------------|
|                          | 4047 Å.<br>Cellofas A Film      | 5461 Å.<br>Gelatin Film |
| Rhodamine B, 6 hr. ...   | 28                              | 22                      |
| 19 hr. ...               | 36                              | 40                      |
| Rhodamine 6GB, 6 hr. ... | 10                              | 8                       |
| 19 hr. ...               | 14                              | 13                      |
| Chance filters used      | { OB10<br>OV1                   | { OGr1<br>ON16          |

The rate at which the B brand fades compared with the 6GB brand is much higher than could be accounted for merely by its different absorption of energy, and it appears that the fading rates of these dyes do genuinely reflect differences in chemical reactivity.

## EFFECT OF VARIATIONS IN DEPTH OF COLOUR

Most of the present tests were made with a constant dye-substrate concentration. It will be clear from a study of the experimental work

reported by Baxter *et al.*<sup>2</sup> that, if there is a marked difference in the slopes of the  $\log t_f - \log(\text{concn.})$  curves for any two dyes examined, the relative difference between their fading rates might alter considerably, and even change sign, with variation in dye concentration. If so, some of the present comparisons would be invalid. This possibility is thought to be unlikely, because—(a) the results for the many different dyes are consistent among themselves; (b) it is unusual for dyes so close in structure as those examined here to have curves of very different slopes, even on different substrates (Baxter, private communication); and (c) a check experiment with the two basic green dyes gave similar results at different concentrations.

#### NOTE ON CRYSTALLISATION IN GELATIN FILMS

Some of the azo dyes, from oxy-L acid, with substituents of negative  $\sigma$ -value appeared to separate out from gelatin when the cast films dried, the intensity of colour decreasing markedly. No chemical reaction is involved, because solutions containing gelatin and dye show a similar change when left to cool and set, which is reversed on reheating; presumably the change is due to crystallisation of dye on cooling. Fading results with these films showed irregular relative rates. Disulphine Green B in gelatin also lost considerable colour intensity on drying, but this dye is not sufficiently close in structure to any of the others to justify comparison of rates.

#### NOTE ON HYDROGEN PEROXIDE OXIDATION OF AZO DYES

The curves of relative degradation rate plotted against  $\sigma$ -value for the reaction of hydrogen peroxide with the R-acid dyes have positive slope (indicating reduction) for acid solutions, and negative slope (indicating oxidation) for alkaline solutions<sup>3</sup>. This change is the reverse of that which might at first sight be anticipated from the known values for the redox potential of hydrogen peroxide solutions<sup>4</sup>, e.g. +0.81 v. at pH 2.0 and +0.30 v. at pH 10.0. These figures indicate a less powerful oxidising action in more alkaline solutions, and towards systems of suitable redox potential hydrogen peroxide may in fact act as a reducing agent in solutions of high pH value, e.g. the potassium ferrocyanide-ferricyanide and the silver-silver halide systems. It is, however, incorrect to consider that azo dye-hydrogen peroxide solutions must behave similarly to these inorganic systems, because the initial reaction with the dye is not ionic; e.g. the primary reaction must be hydrolysis to a phenylhydrazine derivative

and a quinone<sup>5,10</sup>, and the former is probably then oxidised to a diazo compound<sup>6</sup>.

\* \* \*

The authors wish to thank Professor P. D. Ritchie for his interest and encouragement; the International Wool Secretariat and the Wool Textile Research Council for scholarships awarded to J.W.C. and A.E.McE. respectively, and for financial grants towards the cost of materials; Mr. G. Baxter for assistance with some of the experiments; and Ilford Ltd. and Imperial Chemical Industries Ltd. (Nobel and Dyestuffs Divisions) for gifts of materials.

COLOUR CHEMISTRY RESEARCH LABORATORY  
DEPARTMENT OF TECHNICAL CHEMISTRY  
ROYAL TECHNICAL COLLEGE  
GLASGOW C.1

(Received 12th November 1955)

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- 13 Traill, *J.S.D.C.*, **67**, 257 (1951).
- 14 Graham and Statham, *ibid.*, **72** (Sept. 1956).

#### NOTE ADDED IN PROOF

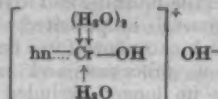
In view of Graham and Statham's observation<sup>14</sup> that the tryptophan residue in wool is oxidised photochemically in presence of fluorescent compounds, it seems possible that further investigation might confirm that tryptophan could in some cases act as a reducing agent in the fading of dyes on proteins (cf. p. 378).

#### ERRATUM

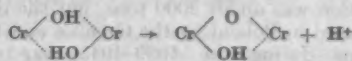
Baxter *et al.* (*J.S.D.C.*, **71**, 228 (May 1955))—Recalculation of experimental data has revealed some errors in the  $t_1$  and  $t_{eq}$  figures given, but the qualitative difference between the two films is unaffected. Column 2 should now read (down) 10.0, 5.2, 2.4, —, —, 4.5, 2.5, —. The  $t_{eq}$  figures are now considered unnecessary for the required comparison.

#### ERRATA

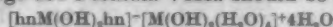
**A Study of Certain Natural Dyes—II.** F. M. Arshid *et al.* (*J.S.D.C.*, **70**, 402–412 (Sept. 1954))—On page 404 (second column) Formula II should be—



On page 405, at the bottom of the first column, the formulation of the "oxolation" process should be—



On page 411 Formula VIIIa should be—





## Notes

### Meetings of Council and Committees

#### July

- Council—4th
- Finance and General Purposes—4th
- Colour Index Editorial Panel—10th and 25th
- Publications—17th
- Terms and Definitions—12th
- Diplomas—4th
- Perkin Executive—5th
- Review of Textile Progress—19th

### Deaths

We regret to report the loss by death of Mr. A. Forsythe and Dr. H. Levinstein.

### Centenary of the Discovery of Magenta

This year marks not only the centenary of Perkin's discovery of Mauve and of its use as a dye but also that of Fuchsine or Magenta (*C.I.* 677) by the Polish chemist Jakub Natanson (*Annalen*, 98, 297 (1856)), who while working in the university laboratory at Dorpat, prepared Magenta by treating aniline with ethylene chloride but failed to recognise its potentialities as a dye. Magenta was prepared for a second time by Hofmann in 1858, who treated aniline with carbon tetrachloride; but it was Verguin's oxidation of aniline with stannic chloride in 1859 which formed the basis of the first industrial manufacture of Magenta and made it the starting point for the manufacture of triphenylmethane derivatives. The commercial exploitation of Verguin's process owed much to the manner in which a Lyons firm of silk dyers, Renard Frères, exploited the possibilities opened up to them by being the first dyers to have the dye made available to them.

C.O.C.

### The Polish Dyemaking Industry

In addition to Natanson, who was the first to prepare Magenta, other Poles who worked on dye chemistry in the late nineteenth and early twentieth centuries included Marcell Nencki (1847-1901), Stanislaw Kostanecki (1860-1910), and Stefan Niementowski (1866-1925). Present-day Poles who, according to the Polish Cultural Institute in London, have contributed greatly to the development of dye chemistry in Poland include Wiktor Lampe, Wacław Lesiański, and Edward Trepka, all professors in Polish universities.

Manufacture of dyes in Poland did not start until 1888, i.e. 32 years after Perkin had founded the industry in England. In 1939 the annual production was about 2000 tons, but the devastation caused in Poland by the German and Russian invasions during the 1939-1945 war reduced production so severely that only a little over 200 tons of dyes was produced in 1945. However,

such were the efforts put into resuscitating the industry that by 1947 production surpassed that of prewar years. During 1950-1955 production of dyes in Poland rose by more than 150%, and over 170 dyes were made which had not previously been made there. In the schemes now being carried out for the further development of the Polish dyemaking industry, particular attention is being paid to the production of fast dyes for wool.

In view of the development of the dyemaking industry in Poland, it is unfortunate that the Society, when compiling the data for the Second Edition of the *Colour Index*, has found that its efforts to secure the co-operation of the Polish dyemaking industry, or indeed to obtain any information at all from authoritative sources about Polish dyes, have met with not the slightest success.

C.O.C.

### Documentary Film on Titanium Pigments

A 36-mm. colour film, *The Titanium Pigment Story*, which runs for 23 min., is available from the British Titan Products Co. Ltd., York, who are also able to provide 16-mm. prints for showing to professional and trade organisations. The film deals with the manufacture of titanium pigments and shows their use in paints, textiles, rubber, plastics, linoleum, vitreous enamels, etc.

C.O.C.

### Flameproof Finish

The mixture of compounds known as phosphorylamide, formed from phosphorus oxychloride and ammonia, was found some years ago to possess excellent flameproofing properties, but suffered from drawbacks in manufacturing difficulties, and in causing weakening and stiffening of the fabric to which it was applied. Improvements in manufacturing technique and methods of purification of phosphorylamide have now been developed, and it is expected to be available at a relatively low cost compared with tetrakis(hydroxymethyl)phosphonium chloride and other organic phosphorus compounds. The application procedure has been modified so that the fabric is not appreciably weakened. Phosphorylamide is applied by padding from dilute aqueous solution followed by drying and curing at 140°C. The treated fabric is said to remain flameproof after 25 launderings. (*Ind. Eng. Chem.*, 48, 22A (March 1956, Part I).)

W.K.R.

### Swiss Patent Law

Under the former Swiss patent law (a) new chemical compounds and (b) new processes which were not purely mechanical, for the improvement of textile fibres, were not patentable. Thus, new dyes and new textile-finishing and -dyeing processes could not, in general, be patented. Under a new patent law, which came into force on 1st January 1956, such new processes and new chemical compounds are no longer excluded from patent



protection, but patents for them will not be granted until an official examination for novelty has been introduced. It is not expected that this will be in operation before 1958. Any applications for patents for chemical compounds or new mechanical processes for improving textiles which have been filed before the official examination will be redated to the date when the examination is introduced.

L.E.J.

### Photographs of Individual Atoms

Individual atoms of tungsten on the surface of a solid were photographed by Erwin Müller at

Pennsylvania State University late in 1955. The atoms were about  $5 \times 10^{-9}$  in. in diameter. He used a field ion microscope developed from the field electron microscope he devised twenty years ago. The new microscope enables much better resolution to be obtained by using ions with a shorter de Broglie wavelength to form the image and by working at the temperature of liquid hydrogen to obtain sharper images. The new method renders visible for the first time the atomic structure of the surface of the tungsten needle. It can therefore be used for the study of surface films.

C.O.C.

## OBITUARY NOTICE

### James Anthony Felce

James Anthony Felce met his death tragically in a road accident on 25th April 1956, at the age of 24. He entered the University of Leeds in October 1950, and his academic record was consistently good. He was awarded the Diploma in Dyeing in 1953, and in the same year gained the First Prize and the Silver Medal in the Final Grade examination in Wool Dyeing of the City and Guilds of London Institute. He graduated with Second Class Honours in Colour Chemistry in June 1954, and at the time of his death was engaged on

research for a higher degree under Professor W. Bradley.

He was keenly interested in Departmental affairs, acting as Honorary Secretary in connection with the Annual Departmental Dinner in 1955. During the 1954-1955 session he served the Society as Honorary Secretary of the Leeds Junior Branch. He was a very likeable fellow of considerable promise, and his untimely death is deeply deplored by his fellow students and the staff of the Department.

C. B. STEVENS

## New Books and Publications

### First World Congress on Surface-active agents Paris: Chambre Syndicale Tramagras. [1956.]

Volume 1 (Sections 1-4), pp. 1-421;

Volume 2 (Sections 5-11), pp. 427-879;

Volume 3 (Sections 12-17), pp. 883-1353.

This congress was held from 30th August to 3rd September 1954 at the Sorbonne. More than 1200 delegates from 32 countries were present; the British delegation with 116 members was numerically the second largest. These were 523 delegates from France, 72 from Germany, 58 from Italy, 57 from Belgium, 51 from Holland, 40 from the U.S.A., 30 from Spain, 22 from Switzerland, etc. The Conference was arranged by the Chambre Syndicale Nationale des Transformateurs de Matières Grasses et Fabricants de Produits Auxiliaires (Tramagras) and a number of French associations and scientific societies.

The programme of the Congress was divided into 17 sections, and altogether 209 papers were read and discussed. The proceedings of the Congress have now been published. As there were no preprints available, it is now possible to read and study each paper in full. In the reports the papers are published in the language in which they were presented (the official languages were French and English), and as the 103 papers by French authors were not translated during the sessions, one would have wished them translated into English (or *vice versa* the 44 English papers into French) in the final report. Each paper is followed by a list of references and a brief (but not verbatim) report of the discussions (mostly in French). At the end

of the report of each section there is a page of conclusions, which summarise the activities of each section and make recommendations for further studies.

The report of each section is printed also as a separate booklet, and as several of the papers are to be abstracted in our *Journal*, it seems sufficient to state the title of each report—

| Section | Title  | Pages | Price<br>(French<br>francs*) |
|---------|--|-------|------------------------------|
| 1       | Physical Chemistry of Surface-active Agents                              | 151   | 440                          |
| 2       | Analysis and Identification of Surface-active Agents                     | 67    | 400                          |
| 3       | Test Methods for Surface-active Agents                                   | 87    | 400                          |
| 4       | Production, Equipment, Materials of Construction, Raw Materials, Methods | 98    | 440                          |
| 5       | Textile Industry, Dyeing, Printing, Bleaching, and Finishing             | 117   | 400                          |
| 6       | Leather, Fur, and Paper Industries                                       | 65    | 360                          |
| 7       | Laundering, Cleaning, Scouring   | 41    | 280                          |
| 8       | Household Cleaning   | 37    | 240                          |
| 9       | Hygiene and Cosmetics  | 78    | 360                          |
| 10      | Pharmaceutical Industry, Medicine, Surgery                               | 71    | 400                          |
| 11      | Mechanical Industry, Metallurgy  | 35    | 280                          |
| 12      | Mines and Ores, Flotation  | 163   | 440                          |
| 13      | Equipment, Transport   | 19    | 200                          |
| 14      | Farming, Food, and Phytopharmaceutical Industries                        | 79    | 400                          |
| 15      | Paints and Varnishes, Resins and Elastomers                              | 43    | 280                          |
| 16      | Civil Engineering, Construction  | 35    | 280                          |
| 17      | Economic and Social Problems   | 55    | 400                          |

\*£1 = 980 French francs

Many of the papers read at the Congress were of fundamental interest. Questions of classification, standardisation, and testing methods were also discussed, and it was resolved that national committees should be formed in each country, to define and classify the materials and products used in the detergent industry, to work out a nomenclature and terminology of such products, their properties and characteristics, and to define methods of analysis, testing, and evaluation. Such national committees should establish a close liaison among

themselves and should co-operate with the International Committee on Detergents. In the United Kingdom the Oils and Fats Group of the Society of Chemical Industry has recently formed a Surface-active Agents Panel (Chairman Mr. R. C. Tarring).

Preparations are well in hand for the Second International Congress of Surface Activity, which will be held in London during 8–12th April 1957, with Sir Eric Rideal, M.B.E., F.R.S., as President.

A. F. KERTESS

## Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., 68, 23 (Jan. 1952), and also, together with symbols and the periodicals abstracted, in the annual index

### I—PLANT; MACHINERY; BUILDINGS

#### Unit Operations in Chemical Engineering—Developments in 1955

*Ind. Eng. Chem.*, 48, 467–559 (March 1956)

#### Fundamentals of Chemical Engineering—Annual Review

*Ind. Eng. Chem.*, 48, 561–684 (March 1956)

#### Applications of Chemical Engineering in the Fine Chemicals Industry

W. C. Peck

*Chem. and Ind.*, 157–163 (10 March 1956)

A brief summary is given of the features essential for chemical plant in the fine chemical industry, especially in connection with contamination by the materials of construction, extraneous material, and residual material from earlier batches. Examples of plant used in grinding, mixing powders and pastes, distillation and evaporation, and sterile processing are given.

C.H.R.

#### Historical Notes on the Wet-processing Industry. XI—Coppers, Kettles, and Vats

S. M. Edelstein

*Amer. Dyestuff Rep.*, 45, 245–249 (23 April 1956)

#### Dominant (Gerber) Yarn-dyeing Machine

R. Vogel

*Textil Praxis*, 11, 393–396 (April 1956)

Details of the Dominant yarn-dyeing machine made by the Gerber works in Crefeld are given with special reference to its use in the application of vat dyes to rayon yarns.

B.K.

#### Photomechanical Engraving of Printing Rollers

A. Franken

*Textil Praxis*, 11, 383–385 (April 1956)

The apparatus used for engraving printing rollers by photomechanical processes is illustrated and described in detail.

B.K.

#### Calendering and Calender Bowls

R. G. Oliver

*Proc. Tech. Sectn. British Paper &*

*Board Makers' Asscn.*, 36, 665–667 (Dec. 1955)

The fundamentals of the calendering process, the effect of the different materials of which bowls are made on the sheet properties, and calender maintenance are discussed.

K.W.

### PATENTS

#### Thread-storing, Thread-advancing Rolls suitable for Washing of Gelled Polyacrylonitrile Filaments

American Cyanamid Co.

USP 2,714,052

#### Loading and Unloading Yarn into and from Dye Vats

B. W. Mills

BP 749,376

A carrier to convey the yarn to and from the vat and support it while in the vat has locating notches which fit into a set of correctly assembled rods for holding the yarn. There are means capable of passing below the rod ends to hold them in place when picked up by the carrier.

C.O.C.

#### Maintaining Travelling Webs or Films in Transverse Alignment

Cellophane Investment Co.

BP 746,808

One edge of the web or film passes over part of a slot in a plate. Light is directed towards the slot so that part of it is reflected by the web and the other part passes through the slot. When alteration in the amount of light passing through the slot occurs, the necessary adjustment in the lateral position of the web is made.

C.O.C.

#### Web-guiding Device

Union Wadding Co.

USP 2,716,026

#### Safety Device for Squeeze Rollers

Bleachers' Asscn.

BP 749,325

A device for detecting minute projections on rollers comprises a feeler which bears against a substantial width of the roller. Electromechanical detecting means mechanically connected to the feeler are electrically connected through a relay circuit to warning, indicating, and/or control means.

C.O.C.

#### Continuous Steaming and Blowing or Decatising Machine

James Bailey (Engineers)

BP 749,084

There are two perforated rollers, steam being blown through one and air drawn or blown through the other. A continuous cloth guide carries the cloth around the steaming roller and then around the cooling roller.

C.O.C.

#### Fabric-folding Machinery

R. B. Gibson

BP 748,832

Cloth, e.g. from a tenter, is fed between a pair of rollers mounted on a swinging arm. The rollers are so moved relatively to one another that one of them contacts the upper fold of a pile of cloth when the arm swings in one direction, and the other roller makes similar contact when the arm goes in the other direction.

C.O.C.

#### Electrostatic Flock Printing Machine

Velvray Corpn.

USP 2,715,585

#### Apparatus for Coating the Edges of Cloth

U.S. Secretary of the Army

USP 2,713,320

#### Practical Aspects of Dyeing in the Burlington Beam Machine (VIII p. 400)

Shrinking Knitted Fabric (X p. 403)

### II—WATER AND EFFLUENTS

#### Ion-exchange Terminology

T. V. Arden

*Chem. and Ind.*, 320 (28 April 1956)

The present multiplicity and confusion of terms in ion-exchange terminology is regretted and, in the interests of correctness and uniformity, several proposals are made. The following terms are recommended—absorbant, absorbate, desorption, development, displacement development, elution development, elution, eluant, eluate, influent, effluent, regenerate(-ion). The abandonment of the following terms is proposed—elutriant, elutriate, regenerated form.

J.W.D.

**Meaning of "Sequestration"**

R. L. Smith

*Chem. and Ind.*, 320-321 (28 April 1956)

The writer draws attention to the absence of a satisfactory definition of *sequestration* and, indeed, of general agreement as to its precise meaning. The following definition is proposed and defended—The removal of the ionic form of the metal by the formation of a chelate soluble in the medium in which the metal was dissolved before chelation or would have been dissolved if it had not been precipitated from it by some ion.

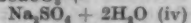
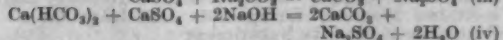
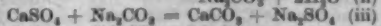
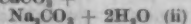
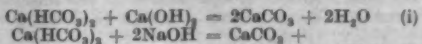
J.W.D.

**Problem of Water in the Manufacture of Leather**

Dr. Riess

*Rev. Tech. Ind. Cuir*, 48, 53-57 (March 1956)

Leathers such as *suedes* often acquire a harsh handle, through the deposition of  $\text{CaCO}_3$  crystals, when they are rinsed with hard water; the following reactions may occur—



The reactants in (i) may originate in the liming of the skin, whilst the  $\text{NaOH}$  in (ii) may result from the hydrolysis of  $\text{Na}_2\text{S}$  used in the unhairing. Pretreatment of a hard water with milk of lime is usually a matter of guesswork and is frequently ineffective. The sequestration of  $\text{Ca}$  by small additions of a polyphosphate (e.g. Calgon) is described and explained. Water so softened may be used in all phases of leather manufacture, and will give *suedes* that are smooth and clear (since  $\text{Fe}$  also is complexed). It also causes slight swelling of the skin and thus aids the penetration of vegetable tannins. Other difficulties in dyeing and dressing due to hard water are avoided.

J.W.D.

**III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS****Kinetic Investigation of the Reaction between Carbon Disulphide and Caustic Soda Solution**

M. Wronski

*Faserforsch. und Textiltech.*, 7, 175-176 (April 1956)

The kinetics of the reactions leading to formation of sodium thiocarbonate and sodium sulphide are discussed and kinetic equations are derived. A linear relationship between reaction time and the logarithm of the ratio of the initial  $\text{NaOH}$  concn. to that at any time is suggested. It is also suggested that the rate of formation of thiocarbonate is initially less than that of sulphide but eventually becomes greater, the amount of sulphide reaching a limiting value. These suggestions are tested and found to be correct.

W.R.M.

**Polymorphism of Alkyl Sulphate Soaps**

J. A. Prins and W. Prins

*Nature*, 177, 535-536 (17 March 1956)

Twelve modifications of the unit cells of recrystallised alkyl sulphate soaps ( $\text{NaSO}_4 \cdot \text{C}_n\text{H}_{2n+1}$ ;  $n = 8, 9, 10, 12, 14, 16$ ) are reported from X-ray measurements. These are thought to be connected with differences in tilt of the hydrocarbon chains and the number of water mol. (from the solvent) associated with the hydrophilic groups.

A.J.

**Interaction between Surface-active Agents and Proteins. I—Precipitates formed by mixing Sodium Alkyl Sulphates and Egg Albumin**

K. Aoki and J. Hori

*Bull. Chem. Soc. Japan*, 29, 104-110 (Jan. 1956)**Structure of Hydrolysed Titanium Dioxide**

C. de Rohden

*Chim. et Ind.*, 75, 287-291 (Feb. 1956)

Titanium dioxide for pigments is prepared by the hydrolysis of soln. resulting from the attack of  $\text{H}_2\text{SO}_4$  on ilmenite. Electron-microscopical examination of the hydrolysate and of the resulting colloidal soln. leads to the following conclusions—(1) The hydrolysate is a gel consisting of very loose flocculates, the size of which varies with the conditions of observation. (2) It has a granular structure, the granules being  $\sim 0.6-0.7 \mu$  in diameter. (3) Each of these granules is an agglutinate of ca. 1000 granules of  $60-75 \text{ m}\mu$  cemented by adsorbed  $\text{SO}_4^{2-}$  ions.

(4) Each of the latter granules contains ca. 20 microcrystals of  $20 \text{ \AA}$ . each, these being the seeds added to the soln. in order to induce hydrolysis. Three electron micrographs are reproduced.

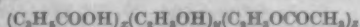
J.W.D.

**PATENTS****Hydrolysed Vinyl Acetate-Crotonic Acid Copolymers as Sizes for Nylon Yarn**

Du Pont Co. of Canada

USP 2,715,590

Hydrolysed vinyl acetate-crotonic acid copolymers of formula—



$x, y$  and  $z$  = numbers;  $y + z : x :: 5.5-19 : 1$ ;  $y/z < 1/9$  have as good sizing property as polyvinyl alcohol and much better adhesion to the fibre, so that addition of other ingredients, e.g. boric acid, is unnecessary.

C.O.C.

**Dilute Aqueous Suspensions of Modified Polyamides—Textile Finishes**

DuP

USP 2,714,075

Stable dilute aqueous suspensions of *N*-alkoxymethylpolyhexamethylene-adipamides are obtained by using as stabilising agent a water-soluble salt of an acid having ionisation constant for the first hydrogen of  $< 1 \times 10^{-5}$ , e.g. ammonium or morpholinium citrate. Such suspensions are used as textile finishes on all types of fibres to impart increased resistance to abrasion and snagging, antistatic properties, dimensional stability, increased water adsorption, improved dye fastness, delustering, anti-slip finish and increased tensile strength.

C.O.C.

**Polymeric  $\beta$ -Propiolactone as Antistatic Agent**

U.S. Secretary of Agriculture

USP 2,715,592

Polymeric  $\beta$ -propiolactone is an excellent antistatic agent for application to wool.

C.O.C.

**Aqueous Propylene Carbonate as Solvent for Acrylonitrile Polymers**

ICI

BP 748,303

Aqueous propylene carbonate is a good solvent for acrylonitrile and grafted acrylonitrile polymers.

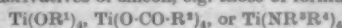
C.O.C.

**Organo Titanium-Silicon Copolymers—Water-repellent Agents and Catalysts for Curing Condensation Resins**

Monsanto

USP 2,716,656

The copolymers of organic derivatives of titanium with organic derivatives of silicon, e.g. those of formula—



( $\text{R}^1$  = monovalent hydrocarbon;  $\text{R}^2$  = Alk, cyclic or acyclic radical or Ar;  $\text{R}^3$  = H, Alk or Ar;  $\text{R}^4$  = Alk or Ar) are useful as curing catalysts for condensation resins, e.g. urea- or melamine-aldehyde resins, and for imparting a water-repellent finish to textiles which is resistant to immersion of 24 hr. or repeated laundering or dry cleaning.

C.O.C.

**Ultraviolet-radiation-absorbing Compounds and their Use in Colour Photography (IX p. 401)****Modern Resinous Tanning Materials, their Properties and Uses (XII p. 406)****Laboratory and Field Exposure Studies of Leather Fungicides (XII p. 406)****Chelate Complexes and the Chelatometric Determination of Calcium and Magnesium Ions (XIV p. 407)****Relative Efficiency of Chelating Agents (XIV p. 407)****IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS****The Genius of Perkin**

L. E. Morris

*Dyer*, 115, 747-764 (11 May 1956)**Perkin and the Dye Industry in Britain**

R. Brightman

*Nature*, 177, 815-821 (5 May 1956)**The Perkin Family of Organic Chemists**

Sir Robert Robinson

*Endeavour*, 15, 92-102 (April 1956)**Synthetic Dyes from the Time of Perkin**

A. Johnson and H. A. Turner

*Dyer*, 115, 765-769 (11 May 1956)



**Catalytic N-Alkylation of Aniline with 1-Propanol**

B. B. Corson and H. Dressler

*J. Org. Chem.*, **21**, 474 (April 1956)

Contrary to Rice and Kohn (*J. Amer. Chem. Soc.*, **77**, 4052 (1955)), U.O.P. nickel catalyst is effective, giving an 80% yield of *N*-*n*-propylaniline in 6 hr. H.E.N.

**Preparation of Substituted Hydrazines. IV—Arylhydrazines by Conventional Methods**

I. M. Hunsberger, E. R. Shaw, J. Fugger, R. Ketcham, and D. Lednicer

*J. Org. Chem.*, **21**, 394–399 (April 1956)

The properties and the preparation of salts of 28 substituted phenylhydrazines, three substituted naphthylhydrazines, 2-hydrazinofluorene (64% yield), 9-phenanthrylhydrazine (28%), 3-hydrazinopyrene (23%, could not be repeated), and 6-quinolylhydrazine (45%) are described. No hydrazine could be obtained from 3-amino-acenaphthene. H.E.N.

**Nitration with the Boron Trifluoride-Nitrogen Tetroxide Complex**

E. R. Ward

*Chem. and Ind.*, 195 (24 March 1956)

The work of Bachman *et al.* on the  $\text{BF}_3\text{-N}_2\text{O}_4$  nitration of naphthalene (*J. Amer. Chem. Soc.*, **77**, 6188 (1955)), in which a 2:1 ratio of 1:5 and 1:8-dinitronaphthalenes is claimed, i.e. the reverse of that usually accepted for  $\text{H}_2\text{SO}_4\text{-HNO}_3$  nitrations at low temp., is shown by critical inspection of the cited experimental data to be incorrect. Previous work on this reaction is briefly summarised. H.H.H.

**Nitration of 2-Naphthylamine-8-sulphonic Acid**

L. Blangey

*Helv. Chim. Acta*, **39**, 977–980 (May 1956)

Apart from the previously obtained 6-nitro epd. (30% yield), the 4- (25%) and 5-isomers (30%) are isolated separately by pptn. of the first as the Mg salt, the second as the Zn salt, and the last as the free acid, purified as the K salt. Identification of the new epd. was carried out by deamination and reduction. The 4-nitro deriv. on reduction gives a diamine which couples with benzenediazonium salts to yield yellow dyes. H.E.N.

**Rate of Reduction of Aromatic Nitro Compounds by Béchamp's Method**

S. Yagi, T. Miyauchi, and C. Y. Yeh

*Bull. Chem. Soc. Japan*, **29**, 194–200 (Jan. 1956)

The reduction of nitrobenzene with iron powder in dil. HCl has been studied using a steel vessel fitted with a variable-speed stirrer. The results indicate that the reaction proceeds mainly on the iron surface providing that the agitation is sufficient to prevent settling of the iron powder. After an induction period the rate of reduction is of zero order and proceeds mainly at the iron-aqueous layer interface, but in the final stage the reaction is of the first order and occurs at both the iron-water and iron-oil interfaces. The proportion of water is important and should be at least six times that of the nitro compound for a smooth reaction. The concn. of chloride ion has little effect between 0.8 and 2.7 *N*.; below 0.8 *N*. the rate decreases rapidly. Other nitro compounds show similar behaviour. A.J.

**Selective Reduction of 2:2':4:4'-Tetranitrodiphenyl**

W. J. Bailey

*J. Org. Chem.*, **21**, 480 (April 1956)

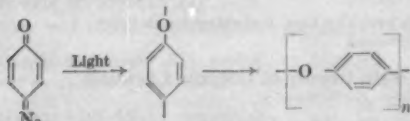
Using methanolic sodium sulphide-bicarbonate a 62% yield of 2:2'-dinitrobenzidine is obtained. H.E.N.

**Photochemistry of Diazo Compounds. VII—Quinone-*p*-diazides and Iminoquinone-diazides**

O. Süs, K. Möller, and H. Heiss

*Annalen*, **598**, 123–138 (April 1956)

The reactions of these epd. are of practical importance in photomechanical processes. The mechanism was thought to be of the type—



This was confirmed by exposure of diazides in presence of aliphatic alcohols, aromatic hydrocarbons, and heterocyclic

epd., the  $\text{N}_2$  group being replaced by OR and Ar. These reactions can be used preparatively with good results, many examples being quoted. H.E.N.

**Effect of Dye Structure on the Dyeing of Orlon 42 with Azo Dyes**

A. J. Pelletier

*Amer. Dyestuff Rep.*, **45**, P 227–P 230 (9 April 1956)

Orlon 42 is dyed with 25 monoazo and disazo dyes, of systematically varying structure. In general, increase in mol. wt. increases the dye affinity in acid dyebaths but not in salt soln. It is possible that an extra azo group may be more influential than mol. wt. alone. Sulphonic acid groups appear to prevent adsorption of monoazo dyes, though if they are correctly positioned there will still be considerable affinity. With disazo dyes, as the number of sulpho groups is increased from one, the salt dyebath exhaustion decreases to a variable degree. The influence of hydroxy groups is not clear. Amino groups present in the molecule, either alone or in excess of sulpho groups, facilitate dyeing. J.W.B.

**Internal Complexes of Azo Compounds. III—Reactions of Copper Salts with some *o*-Hydroxy and *o*-Amino Monoazo Compounds containing an *o*-Alkoxy or *o*-Aryloxy Substituent**

V. I. Mur

*J. Gen. Chem. U.S.S.R.*, **26**, 384–389 (Feb. 1956)

Previous investigations (see J.S.D.C., **71**, 472 (1955)) on *o*-hydroxy- and *o*-amino-azo epd. containing *o*-methoxyl are extended to *o*-ethoxy and *o*-phenoxy epd. Like the methoxy epd., the ethoxy epd. give 1:1 copper complexes when treated with alcoholic  $\text{CuCl}_2$ , but the phenoxy epd. do not give complexes under these conditions, though they give the usual 1:2 complexes when treated with Cu salts under other conditions. A.E.S.

**Relationship between Structure, Colour, and Substantivity of Dyes derived from Benzanilide**

B. M. Krasovitskii, B. I. Ostrovskaya, and D. G. Peroyaslava

*Doklady Akad. Nauk S.S.S.R.*, **106**, 72–75 (1 Jan. 1956)

The colour and the substantivity on cotton of 16 benzanilide deriv. (mainly azo epd.) are examined, and attempts are made to correlate these properties with the structures of the epd., particularly with respect to linearity, coplanarity, conjugation, and opportunities for hydrogen bonding with cellulose. Deep colour and high substantivity are favoured by the linearity found in e.g. 4- and 4'-phenylazo- and 4:4'-bisphenylazo-benzanilides and by the possibility of the existence of the substance in a tautomeric imidol form having an unbroken conjugated chain (as in the phenylazo epd. cited). A.E.S.

**Physical Properties of Aminoazobenzene Dyes. II—Further Studies of their Basicity**

E. Sawicki and D. Gerber

*J. Org. Chem.*, **21**, 410–412 (April 1956)

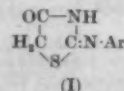
The basicities in 50% ethanol of 34 dyes, 19 prepared for the first time, are determined, some infrared absorption maxima being recorded also. H.E.N.

**Preparation of 5-*p*-Sulphonamidophenylazo-2-aryl-imino-4-thiazolidones**

G. N. Mohapatra and M. K. Rout

*J. Indian Chem. Soc.*, **33**, 17–20 (Jan. 1956)

2-Arylimino-4-thiazolidones (I), where the aryl group is phenyl, tolyl, nitrophenyl, chlorophenyl, carboxyphenyl, and naphthyl, have been prepared by condensing the arylthiourea with monochloroacetic acid in the presence of anhydrous sodium acetate, or with ethyl monochloroacetate and sodium ethoxide. These have been coupled with diazotised sulphanilamide to give the corresponding 5-azo compounds.



(I)

C.H.R.

**Thiazoles. XXVII—2-Hydrazinothiazoles**

H. Beyer, C. Bischoff, and G. Wolter

*Chem. Ber.*, **89**, 1095–1099 (May 1956)**Formazans and Tetrazens—II**

H. Hauptmann and A. C. de M. Périassé

*Chem. Ber.*, **89**, 1081–1094 (May 1956)



**5:6-Benzoquinoline-2-aldehyde**

W. Matthes and W. Sauermilch

*Chem. Ber.*, **89**, 1183-1184 (May 1956)

5:6-Benzoquinoline, obtained in 64% yield from 2-naphthylamine, is oxidised with  $\text{SeO}_2$  in toluene in 48% yield. H.E.N.

**Systematisation of Ultraviolet Absorption. III—Polycyclic Compounds containing the Naphthalene Nucleus**

H. Dannenberg and D. Dannenberg-von Dresler

*Chem. Ber.*, **89**, 1326-1333 (May 1956)**Reactions of Pararosaniline, Aniline Blue, and their Carbinols with Acids**

V. F. Lavrushin, T. M. Shmaeva, and I. M. Nikolaeva

*Doklady Akad. Nauk S.S.S.R.*, **105**, 492-495 (21 Nov. 1955)

The conversion of the colourless carbinol of a triphenylmethane dye into the coloured dye by the action of acid has been widely attributed to the formation of a conjugated cation that is represented as a substituted ammonium salt. Further treatment of the dye, e.g. pararosaniline (*C.I.* 676) in alcoholic soln., with acid results in salt formation at another N atom and a consequent colour change. In Aniline Blue (triphenylated pararosaniline, *C.I.* 688) the nitrogen should be much less basic than in pararosaniline, and in fact a very much higher concn. of acid is required to change the colour of an alcoholic soln. of the dye. However, when weak acids (acetic, carbonic, boric) are added to alcoholic soln. of the carbinols of these two dyes, in both cases the colour of the dye appears (confirmed quantitatively by the absorption spectra); i.e. the basicities of the carbinols are high and do not differ greatly, in spite of the very low basicity of the diphenylamine residues of Aniline Blue. It is concluded that these dyes are carbonium salts and that their colour has essentially the same origin as the colour of salts formed by treating non-nitrogenous aromatic carbinols with acid, although these latter salts are usually much more readily hydrolysed than triphenylmethane dyes. A.E.S.

**Spectrophotometry of Dyes. I—Methyl Green.****II—Pyronine**

P. B. Rottier

*Stain Technol.*, **28**, 265-273 (1953);*Chem. Abs.*, **50**, 6797 (10 May 1956)

Absorption peaks of seven samples of Methyl Green showed that two different types of the dye were present. This offers means of differentiating between heptamethyl and hexamethylethyl derivatives of pararosaniline. Pyronines Y and B are readily differentiated by comparing their absorption maxima. C.O.C.

**Peroxidase Action. X—Oxidation of Phenols**

H. Booth and B. C. Saunders

*J.C.S.*, 940-948 (April 1956)

Hydrogen peroxide oxidation of mesitol in the presence of peroxidase gives 4-hydroxy-3:5-dimethylbenzyl alcohol, 4-hydroxy-3:5-dimethylbenzaldehyde, and 2:6-dimethylbenzoquinone. The reaction involves the stepwise oxidation of the methyl group *para* to the OH group; durenol is oxidised mainly to 4:4'-dihydroxy-2:3:5:6:2':3':5':6'-octamethyldiphenyl with a trace of duroquinone. Guaiacol is rapidly oxidised to a brown-red solid which contains 2:2'-dihydroxy-3:3'-dimethoxydiphenyl. H.H.H.

**Effects of Substituents. III—Order of Reactivity to Radicals of Active Methylene Groups**

S. Hünig and G. Feilstöcker

*Annalen*, **598**, 105-122 (April 1956)

A  $10^{-4}$  M. soln. of Wurster's Red in 80% methanol brought to pH 3.3 with acetic acid, which is stable at 40°C. for 16 hr., was used as reagent. Reasons are given why the reaction should proceed by a four-step radical mechanism. The activating effects of X and Y in the methylene epd.  $\text{X-CH}_2\text{-Y}$  are essentially additive in the order  $\text{NO}_2 > \text{CN} > \text{CH}_3\text{CO} > \text{C}_6\text{H}_5 > \text{COOH} > \text{COOR} > \text{CO-NH}_2 > \text{SO}_2\text{CH}_3$  ( $\text{SO}_2\text{CH}_3$ ). It is of interest to compare this order with that obtained for an ionic reaction, viz. that with *p*-nitrobenzenediazonium salts:  $\text{NO}_2 > \text{CO-CH}_3 > \text{CN} > \text{COOR} > \text{CO-NH}_2 > \text{COOH} > \text{SO}_2\text{CH}_3 > \text{SO-CH}_3 > \text{C}_6\text{H}_5$ . Phenyl residues favour greatly a radical reaction but do not increase the acidity of a epd., whereas sulphonyl groups do the opposite. H.E.N.

**Constitution and Light Absorption. VIII—Preparation and Light Absorption of Dimethylpolyenes**

F. Bohlmann and H.-J. Mannhardt

*Chem. Ber.*, **89**, 1307-1315 (May 1956)**Cyanine Dyes**

W. Franke and W. Ried

*Compt. rend. 27 Congr. intern. Chim. ind.*(Bruxelles), **3** (1954):*Industrie chim. belge*, **20** (Spec. No.), 605-607 (1955):*Chem. Abs.*, **50**, 6229 (10 May 1956)

2-(Cyanomethyl)benzimidazole (prepared from *o*- $\text{C}_6\text{H}_4(\text{NH}_2)_2$  and  $\text{CN-CH}_2\text{-CO-NH}_2$ ) or its nuclear- or N-substituted derivatives react with *p*- $\text{NO-C}_6\text{H}_4\text{-NR}_2$  to form anils and with  $\text{R}_2\text{N-C}_6\text{H}_4\text{-CHO}$  to form styryl dyes which act as supersensitisers when used with other sensitising dyes. Cyanine and merocyanine dyes having a CN group on the carbon  $\alpha$  to the benzimidazole ring may also be obtained. Other substituents may be attached to the methin chain. The effect of such variations on light absorption agrees with that expected for cyanine dyes. C.O.C.

**Chemistry of Cyanine Dyes. VII—3:3'-Di-*p*-tolyl-6:6'-dimethylthiacyanines**

I. K. Ushenko

*Ukrain. khim. zhur.*, **20**, 384-389 (1954):*Chem. Abs.*, **50**, 6050 (25 April 1956)

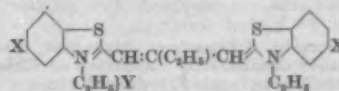
Treating 2-*p*-tolylamino-5-methylphenylthiol with chloroanhydrides of mono- and di-basic acids gave a series of quaternary salts, from which were obtained thiacyanines and *p*-dimethylaminostyryls. C.O.C.

**Absorption and Sensitisation Spectra of 5:5'-Dihaloethiacyanines**

S. V. Natanson

*Doklady Akad. Nauk S.S.S.R.*, **106**, 497-500 (21 Jan. 1956)

Spectra (absorption or reflection) of dyes of formula—



(X = F, Cl, Br, or I; Y = anion) are determined in aq. soln., in gelatin soln. and gels, as films on glass, in gelatin films on glass, in gelatin-AgBr emulsions with and without addition of surface-active agents, and in gelatin-free suspensions and sols of AgBr. Intense J bands indicative of the polymerisation of the dye appear even in dil. aq. soln. and are particularly notable for the chloro and bromo dyes, which, in the adsorbed condition, give also a second J band, apparently due to a macromolecular polymer whose formation is greatly dependent on the solvent and the nature of the surface of the adsorbent. A.E.S.

**Effect of Hydrogen Bonding on the Fluorescence of  $\pi$ -Electron Systems**

N. Mataga, Y. Kaifu, and M. Koizumi

*Bull. Chem. Soc. Japan*, **29**, 115-122 (Jan. 1956)

The addition of proton acceptors (e.g. butyl acetate) to  $\alpha$ - and  $\beta$ -naphthols in *n*-hexane soln. causes a red shift and increased intensity in both the absorption and fluorescence spectra, which is thought to be due to hydrogen bonding. In agreement with this, no shift or intensity changes occur with methyl  $\beta$ -naphthyl ether. Ethyl chloroacetate is exceptional in producing a diminution in intensity of fluorescence. Consideration of the relative intensities of fluorescence of a number of soln. of  $\beta$ -naphthol and proton acceptors suggests that a new equilibrium is set up between free and hydrogen-bonded  $\beta$ -naphthol in the excited state. The mechanism of hydrogen bonding is discussed, and it is concluded that the observed enhanced stability of the hydrogen bond in the excited state is due to increased electron migration. A.J.

**Partly Hydrogenated 1:2:5:6-Dibenzoaceperinaphthanes and their Attempted Dehydrogenation**

H. Dannenberg and D. Dannenberg-von Dresler

*Chem. Ber.*, **89**, 1316-1325 (May 1956)**Fulvenes and Thermochromic Ethylenes. XXX—Experiments in the 2:3-Diaryllindone Series**

E. D. Bergmann

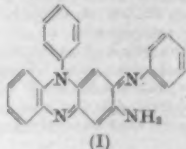
*J. Org. Chem.*, **21**, 461-484 (April 1956)

**Oxidation of Derivatives of *o*-Phenylenediamine**

V. C. Barry, J. G. Belton, J. F. O'Sullivan, and D. Twomey  
**I—Isomeric Phenazine Pigments obtained by Oxidation of 2-Aminodiphenylamine Hydrochloride**

*J.C.S.*, 888-893 (April 1956)

The crude oxidation product from the interaction of 2-aminodiphenylamine hydrochloride and *p*-benzoquinone, after purification on alumina, affords the known 2-anilino-3:5-dihydro-3-imino-5-phenylphenazine (anilino-*apo*-safranin) and its hitherto unknown isomer, 2-amino-3:5-dihydro-5-phenyl-3-phenyliminophenazine (I), both in 5-10% yield.



**II—Phenazine Pigments obtained from *N*-Alkyl-, *N*-cycloalkyl-, *N*-Alkylphenyl-, and *N*-Alkoxyphenyl-*o*-phenylenediamine Hydrochloride**

*Ibid.*, 893-895

The oxidative condensation reaction of I (cf. preceding abstract) when applied to the title deriv. of *o*-phenylenediamine gives corresponding pairs (cf. I) of isomeric phenazine pigments (named on analogy the 2- and the 3-isomers respectively). From *N*-alkylphenyl- or *N*-alkoxyphenyl-*o*-phenylenediamines, the 3-isomer preponderates in the quinone and the 2-isomer in the  $\text{FeCl}_3$  oxidation product. *N*-Alkyl- or *N*-cycloalkyl-*o*-phenylenediamines, however, give only the 3-isomer, whereas *N*-*o*-ethoxyphenyl-*o*-phenylenediamine affords only the 2-isomer, by either oxidation process. Oxidation of *N*-*p*-ethoxyphenyl-*o*-phenylenediamine hydrochloride with quinone gives the 3- and with  $\text{FeCl}_3$  the 2-isomer.

**III—Isomeric Phenazine Pigments and *apo*-Safranones obtained by Oxidation of Chlorinated 2-Aminodiphenylamine Hydrochlorides**

*Ibid.*, 896-899

Oxidation of chlorinated 2-aminodiphenylamine hydrochlorides by  $\text{FeCl}_3$  and by *p*-benzoquinone results in the elimination of chlorine when the latter is present in the 4- or the 5-position, or in both. The products of oxidation are chlorinated anilino-*apo*-safranones or the corresponding chlorinated isomeric phenazines (cf. I), or in some cases, a mixture of the two isomers. The *p*-benzoquinone oxidation also affords a third type of phenazine, viz. chlorinated *apo*-safranones, formed by condensation of the quinone with the diphenylamine deriv. No generalisation as to the mode of elimination of chlorine can yet be made.

H.H.H.

**Polarographic Investigation of Fur Dyes and their Oxidation Products**

G. Sandberg

*J.S.D.C.*, 72, 227-238 (May 1956)

The effect of pH on the oxidation of *p*-phenylenediamine has been investigated. It has been found that the maximum yield of one of the oxidation products—benzoquinone-1:4-bis-(2:5-diaminoanil)—is obtained in a pH interval at which acidity has a marked effect on the state of aggregation of this compound. A change of pH from 6.0 to 7.3 causes its diffusion coefficient to change from  $4.8 \times 10^{-6}$  to  $1.0 \times 10^{-6}$  sq.cm./sec. As this pH interval covers that used in technical fur dyeing, even small fluctuations in pH have an intricate effect on the diffusion properties of this oxidation product, and therefore also on fur dyeing. Absorption spectroscopy indicates qualitative differences between oxidation products formed at different pH values. There is equivalence between the shifts of position and changes of shape of the absorption bands and the observed changes in slope of the curves of half-wave potential against pH for the different products. Catalase in the presence of hydrogen peroxide accelerates the oxidative condensation of benzoquinone-1:4-bis-(2:5-diaminoanil). This change can thus be effected very rapidly without applying the usual chromium or copper salts. Some applications of vertical paper electrophoresis are given, including separation and identification of oxidation products formed under different conditions, and also

demonstration of differences between the dissociation constants of different nitro derivatives. Besides the use of the analytical results in determining the percentages of *p*-phenylenediamine and related compounds in batches from different suppliers, polarography provides a valuable means of determining exhaustion curves in dyebaths, usually comprising a mixture of five or six oxidisable, reducible, and non-reducible substances, where colorimetric methods would meet with difficulties. Hydrogen peroxide, which is always present, is eliminated by the addition of catalase to the different samples of dye liquor.

AUTHOR

**3:6-Dichloro-1:4-quinocarbazole—Yellowish-brown Vat Dye**

K. Sugimoto

*Proc. Japan Acad.*, 31, 300-304 (1955);

*Chem. Abs.*, 50, 6049 (25 April 1956)

Carbazole (20 g.) in acetic acid (75 c.c.) and acetic anhydride (75 c.c.) treated with  $\text{SO}_2\text{Cl}_2$  (21 g.) for 11 hr. at 5-8°C. with stirring yields 3:6-dichlorocarbazole (16 g.). To this (40 g.) in acetic acid (250 c.c.) conc.  $\text{HNO}_3$  (13.2 c.c.) in acetic acid is added dropwise for 11 hr. at < 60°C. to yield 4-nitro-3:6-dichlorocarbazole (39 g.). This (5 g.) in a solution of  $\text{SnCl}_4$  (10 g.) in ethyl alcohol (100 c.c.) is saturated with dry HCl and refluxed for 5 hr. Water is added, the mixture stood overnight, filtered, the ppt. treated with NaOH, extracted with ether, and the extract evaporated to yield 4-amino-3:6-dichlorocarbazole. This (6 g.) is treated with acetic anhydride (30 c.c.) to yield 4-acetamido-9-acetyl-3:6-dichlorocarbazole, which is then treated dropwise with a mixture of  $\text{HNO}_3$  (1.5 c.c.) and acetic acid (2 c.c.) for 1 hr. and filtered to yield 4-acetamido-1-nitro-3:6-dichlorocarbazole (6.5 g.). This (1 g.) is refluxed for 2 hr. in a solution of  $\text{SnCl}_4$  (10 g.) in ethyl alcohol (30 c.c.) saturated with dry HCl, filtered, the ppt. treated with NaOH, extracted with ether, and the extract evaporated to yield 1:4-diamino-3:6-dichlorocarbazole (0.4 g.). This is converted into the dihydrochloride, and 1 g. is refluxed for 2 min. with acetic acid (10 c.c.) and conc.  $\text{HNO}_3$  (0.4 c.c.) to yield 0.25 g. of the yellowish-brown vat dye 3:6-dichloro-1:4-quinocarbazole. C.O.C.

**Alleged Activity of Silver Perchlorate as a Catalyst for the Formation of Phthalides from *o*-Benzoylbenzoic Acid**

H. Burton and D. A. Munday

*Chem. and Ind.*, 316 (28 April 1956)

If  $\text{AgClO}_4$  is carefully freed from  $\text{HClO}_4$ , its effect as a catalyst for the formation of phthalides from *o*-benzoylbenzoic acid is negligible. On the other hand,  $\text{HClO}_4$ , either in anhyd. form in acetic acid-nitromethane soln., or as the 72% azeotrope, is a very efficient catalyst even at room temp. J.W.D.

**Structure of Indigoid Dyes**

H. von Eller

**I—Crystallographic Study Techniques and Methods**

**II—Crystal Structure of Selenoindigo**

**III—Crystal Structure of Indigo**

**IV—Crystal Structure of Thioindigo**

*Bull. Soc. chim. France*, 1426-1444 (1955)

Crystallographic data, tables of calculated and observed structure factors, and diagrams of molecular structures are presented.

**V—Comparison of the Structures of Indigo, Thioindigo, and Selenoindigo**

*Ibid.*, 1444-1449;

*Chem. Abs.*, 50, 6131 (10 May 1956)

In the crystal forms studied, selenoindigo is more closely related to indigo than to thioindigo. All three, however, have analogous distortions of the molecular plane and bonds or interactions between the ketonic O and the dissimilar atom opposite it in the same molecule. Comparison, applicable to the crystalline state only, is made between the various postulated molecular structures for these dyes and the structures indicated by the results of this work. C.O.C.

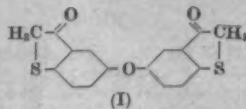
**Indigoid Dyes. XVII—Thioindigoid Dyes derived from the *pp*'-Disulphonic Acid of Diphenyl Ether**

P. C. Dutta and D. Mandal

*J. Indian Chem. Soc.*, 33, 54-56 (Jan. 1956)

The disulphonic acid has been converted to the corresponding 4:4'-bisthioglycollic acid via the mercapto

compound. This on cyclising has given the thioindigoid compound (I), which has been condensed with  $\alpha$ -diketones such as isatin, acenaphthaquinone, phenanthrenequinone, and acenanthraquinone to give thioindigoid dyes. These give reds, violets, and browns on cotton which are deeper in colour than the corresponding dyes from 4:4'-disulphodiphenylmethane reported earlier.



(I)

C.H.R.

#### Action of Alkaline Agents on Quercetin

G. Zwingelstein and J. Jovanetean

*Compt. rend. Acad. Sci. Paris*, 240, 981-983 (1955)

#### Preparation and Configuration of Flavan-3:4-diol

R. Bognár and M. Rákosi

*Chem. and Ind.*, 188 (24 March 1956)

#### Survey of Anthocyanins from Indian Sources—II

J. N. Sharma and T. R. Seshadri

*J. Sci. Ind. Research (India)*, 14B, 211-214 (1955)

An account of the anthocyanin content of 23 Indian plants.

C.O.C.

#### Cyclamen Colours and Pigments

P. Werckmeister

*Saatchi-Wirtsch.*, 7, 339-340 (1955); 8, 7-8 (1956);*Chem. Abs.*, 50, 6593 (10 May 1956)

Review of the chemistry of the anthocyanin pigments present in various *Cyclamen* species. 10 references.

C.O.C.

#### Trolliflor, a Second Pigment from the Flowers of *Trollium europaeum*

M. Lippert and P. Karrer

*Helv. Chim. Acta*, 39, 698-701 (May 1956)

Like trollixanthin, trolliflor is a carotenoid epoxide, containing one additional atom of oxygen, as it has the empirical formula  $C_{40}H_{58}O_3$ . Acid isomerises it to the furanoid oxide trolliflavin.

H.E.N.

#### Carotenoid Syntheses

##### XVIII—An $\epsilon$ , $\epsilon$ -2:2'-Dimethylcarotene

R. Entschel, C. H. Eugster, and P. Karrer

*Helv. Chim. Acta*, 39, 686-690 (May 1956)

##### XIX—A 6:7:6:7'-Tetrahydrolycopen and its Relationship to Neurosporene

C. H. Eugster, E. Linner, A. H. Trivedi, and P. Karrer

*Ibid.*, 690-698

#### Trichosiderin, a Pigment from Human Red Hair

N. A. Barnicoat

*Nature*, 177, 528-529 (17 March 1956)

Sodium hydroxide soln. (0.1 N.) extracts from red hair an orange-yellow precursor of trichosiderin, which yields the pigment on heating in acid. Stronger NaOH causes partial destruction of the precursor. Non-red hairs give a pale yellow alkali extract which does not yield trichosiderin on heating in acid.

A.J.

#### Allomerisation of Chlorophyll

L. G. Johnston and W. F. Watson

*J.C.S.*, 1203-1212 (May 1956)

"Allomerisation" is the term applied to the autoxidation of chlorophyll in alcoholic soln. which precludes it from showing a positive Molisch phase test—the transient brown coloration on addition of alcoholic KOH. Allomerisation is associated with an uptake of oxygen equimolar with the chlorophyll present, and is shown to be influenced by the solvent, the concn. of the chlorophyll, and dissolved salts ( $NaCl$ ,  $NaBr$ ,  $NaI$ ,  $CsBr$ ,  $CsI$ ,  $MgCl_2$ ,  $MgBr_2$ ,  $MgI_2$ ,  $BaCl_2$ ,  $BaBr_2$ ,  $BaI_2$ ,  $LaCl_3$ ). The absorption spectra of the products are reported, and the mechanism of allomerisation is briefly discussed.

H.H.H.

#### Theoretical Studies of the Macrocyclic Pigments.

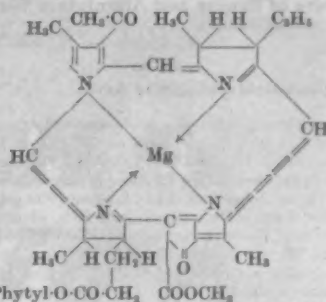
##### I—Structure of Bacteriochlorophyll

J. R. Barnard and L. M. Jackman

*J.C.S.*, 1172-1178 (May 1956)

Molecular-orbital calculations with respect to chlorin (I) and a tetrahydroporphin (II) of  $C_{4v}$  symmetry indicate that the predicted centres of gravity for the longest-wavelength transitions lie in the order: II < porphyrin < I <  $D_{4h}$  tetrahydroporphin. This is in agreement with spectra in the tetraphenylporphyrin series. It follows that bacteriochlorophyll is related to the tetrahydroporphin of  $D_{4h}$

symmetry in which the two pairs of H atoms are located on opposite pyrrole rings, and support for the following structure is thus afforded—

Phetyl-O-CO-CH<sub>2</sub> COOCH<sub>3</sub>

H.H.H.

#### Porphyrin-Metal Complexes in Petroleum Stocks

H. N. Dunning and N. A. Rabon

*Ind. Eng. Chem.*, 48, 951-955 (May 1956)

Very stable vanadium- and nickel-porphyrin complexes have been isolated from the propane-deasphalted raffinate of an Oklahoma crude oil.

C.J.W.H.

#### Pigments for Textiles

G. F. Jones

*Amer. Dyestuff Rep.*, 45, P 263-P 271 (23 April 1956)

The applications and desirable properties of pigments in textiles are briefly described, and advantages and disadvantages of nearly 50 inorganic and organic pigments are listed, with the formulae of the 34 organic types.

J.W.B.

#### Effect of Pigments on the Curing of Silicone Resins

W. Krauss and R. Kubens

*Deut. Farben-Z.*, 10, 1-7 (1956);*Chem. Abs.*, 50, 6811 (10 May 1956)

Pigments used in thermoplastic silicone resins dip-coated on to steel panels and baked fell into two groups. The first group (lampblack, Silcar, Aerosil, talc) did not affect the action of metallic driers; while the second group (inorganic oxides and sulphides) delayed drying, part of this action at least resulting from soluble components present in the pigments.

C.O.C.

#### Modern Application of Luminescent Materials

A. H. McKeag

*Industrial Chemist*, 32, 105-110 (March-April 1956)

A survey of the chemical and physical properties of phosphors, theoretical aspects of luminescence, and the application of phosphors to discharge lamps.

C.O.C.

#### Effect of Thermal Conditions on the Hue of the Red Iron Oxide obtained from Copperas—I

L. N. Uspenskaya, A. G. Bergman, and V. Y. Rudin

*J. Appl. Chem. U.S.S.R.*, 28, 1006-1009 (Sept. 1955);*Chem. Abs.*, 50, 6067 (25 April 1956)

Effect of temperature (600-800°C.), duration of heating (2-10 hr.), drying and not drying before heating, and presence of excess of  $O_2$  on the hue of the pigment produced from  $FeSO_4 \cdot 7H_2O$  was studied. A product containing 6% of soluble Fe and 95-96% of  $Fe_2O_3$  was obtained in 9-10 hr. at 700°C. and in 6-8 hr. at 780°C. The pigment was lighter and brighter when predried  $FeSO_4$  was heated at 750°C. in excess of  $O_2$ . Higher temperatures and longer heating darkened it even when it contained < 96%  $Fe_2O_3$ . Microscopic and X-ray analyses showed only  $\alpha$ - $Fe_2O_3$ , no  $\gamma$ - $Fe_2O_3$  or  $Fe_3O_4$  being formed even in the darker pigments.

C.O.C.

#### Chemistry of the Chromium Oxide Pigments

H. E. Weisburg

*Paint. Ind. Mag.*, 71, (2), 11-17, 41 (1956);*Chem. Abs.*, 50, 6810 (10 May 1956)

Results obtained through a theoretical and statistical approach to the problems of research on chromium oxides.

C.O.C.

#### Orpiment and Realgar in Chinese Technology and Tradition

E. H. Schafer

*J. Amer. Oriental Soc.*, 75, 73-89 (1955);*Chem. Abs.*, 50, 5342 (25 April 1956)

A general survey and discussion of the name, origin, properties, and occurrence of the two As sulphide minerals.



Their use as paint pigments and in early pyrotechnics both in China and in Europe is described. 228 references.

C.O.C.

### Manufacture of Bronze and Aluminium Pigments

J. Konrad and N. Dubay

*Chim. Peintures*, 18, 355-357 (1955)

PATENTS

### Anthraquinonetetrasulphonic Acids

FBy

BP 749,656

Anthraquinone- $\beta$ -sulphonic acids are further sulphonated in presence of < 2% Hg salt and excess  $\text{SO}_2$  at 130-200°C. to yield the 1:3:5:7-, the 1:3:6:8-tetrasulphonic acid, or a mixture of them, according to the starting material. Thus sodium anthraquinone-2:6-disulphonate (500 g.) is heated at 150°C. for 4 hr. with a mixture of 25% oleum (1400 g.) and 65% oleum (500 g.) containing  $\text{HgSO}_4$  (25 g.). After diluting (10 litres water), adding  $\text{HCl}$  (100 g., sp.gr. 1.19), and decolourising with  $\text{NaClO}_2$ ,  $\text{NaCl}$  (1200 g.) is added. Pure sodium anthraquinone-1:3:5:7-tetrasulphonate separates on cooling.

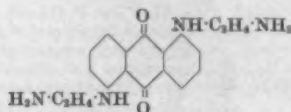
R.K.F.

### Arylamino-aminoalkyl Compounds

Ciba

BP 748,715

A halogenoaryl derivative free from  $\text{NO}_2$  is condensed with an aliphatic primary diamine of 2-6 C to give arylamino-aminoalkyl dye intermediates. Thus 1:5-dichloro-anthraquinone is heated in excess of ethylenediamine for 24 hr. at 40-50°C. to produce—



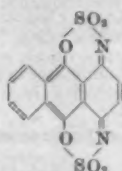
R.K.F.

### 1:4-Diaminoanthracene Derivatives

FBy

BP 749,677

The product of GP 268,592—



obtained by treating 1:4-diaminoanthraquinone with fuming  $\text{H}_2\text{SO}_4$  is converted to 2- and/or 3-substituted 1:4-diaminoanthraquinones by reaction with an alkali-metal bisulphite, an alkali-metal cyanide, a primary or secondary amine, a thiol, or a diene. Thus, by heating with aq. KCN at 30-40°C. the corresponding 2-CN derivative is formed, which by oxidising in  $\text{Na}_2\text{Cr}_2\text{O}_7$ -acetic acid and saponifying with  $\text{H}_2\text{SO}_4$  is converted to 2-carbamyl-1:4-diaminoanthraquinone.

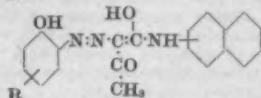
R.K.F.

### Yellow Metal(Cobalt)-complex Monoazo Dyes for Wool, Nylon, etc.

S

BP 748,435

The complexes obtained from 1 atom of Co and 2 mol. of monoazo compounds—



(R =  $\text{SO}_2\text{Alk}$  or a sulphonyl group which may carry Alk, hydroxyalkyl, or methoxyalkyl on the N atom; the naphthalene nucleus may be partly or fully hydrogenated) dye wool, nylon, etc. yellow from neutral or weakly acid baths. Thus diazotised 2-aminophenol-4-sulphonamide is coupled with N-acetoacetyl- $\alpha$ -naphthylamine in presence of  $\text{NaOH}$  and  $\text{Na}_2\text{CO}_3$ , and the monoazo dye so formed is heated with aq. cobalt acetate at 85-90°C. under alkaline conditions to give the cobalt complex.

E.S.

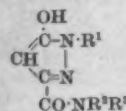
### Metal(Chromium)-complex Monoazo Dyes for Wool

Ciba

BP 749,826

The metal complexes containing 1 atom of Cr to 1 mol. of a monoazo dye formed by coupling a diazotised

o-aminophenol- or o-aminonaphthol-sulphonic acid with a 5-pyrazolone-3-carboxyamido—



( $\text{R}^1$  and  $\text{R}^2$  = H or Alk;  $\text{R}^3$  = Alk, cycloalkyl, aralkyl, or aryl of > 2 C, or  $\text{R}^2$  and  $\text{R}^3$  together = a heterocyclic residue) dye wool reds and bordeaux from sulphuric acid baths. The amides are prepared from the pyrazolone-carboxylic acids by treatment in inert solvents with o.g.  $\text{PCl}_5$  and subsequent or simultaneous condensation with a suitable amine. Thus diazotised 2-amino-4-chlorophenol-6-sulphonic acid is coupled with an alkaline soln. of 5-pyrazolone-3-carbo-n-butylamide, and the monoazo compound so formed is boiled with aq.  $\text{Cr}_2(\text{SO}_4)_3$ . The product dyes wool blue-red.

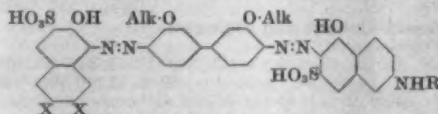
E.S.

### Blue Metal(Copper)-complex Disazo Direct Dyes

ICI

BP 748,611

The copper complexes derived from disazo dyes—



(Alk =  $\text{CH}_3$  or  $\text{C}_2\text{H}_5$ ; one X = H, the other X =  $\text{SO}_3\text{H}$ ; R = benzoyl or phenyl either of which may be substituted by Hal,  $\text{CH}_3$ ,  $\text{CF}_3$ , or O-Alk) are blue direct dyes, redder and faster to resin finishes than the similar dyes of BP 352,956 (J.S.D.C., 47, 360 (1931)). Thus tetrazotised dianisidine is coupled first with a solution of 1 mol. of R salt in aq.  $\text{Na}_2\text{CO}_3$  and then with a solution of 1 mol. of N-benzoyl-J acid in ammoniacal pyridine. The disazo compound so formed is heated at 55°C. with aq. copper sulphate, ammonia, and diethylamine for 18 hr. to give the copper complex.

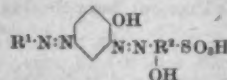
E.S.

### Neutral-dyeing Metal(Chromium)-complex Disazo Dyes for Wool, Nylon, etc.

S

BP 749,142

Metal complexes containing 1 atom of Cr to 2 mol. of disazo compounds—



( $\text{R}^1$  = aryl of benzene or naphthalene series;  $\text{R}^2$  = residue of ortho-coupling naphthol or keto-methylene compound) dye wool, nylon, etc. from neutral or weakly acid baths. Thus, 3-amino-4-hydroxyazobenzene is diazotised and coupled with 2-naphthol-4-sulphonic acid, and the disazo compound so formed is heated at 100°C. in formamide with ammonium chromium sulphate, to give the complex, which dyes wool, silk and nylon grey.

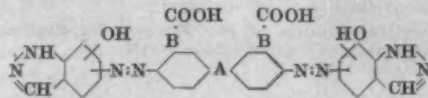
E.S.

### Red to Blue Copperable Dis- and Tri-azo Direct Dyes derived from Hydroxyindazoles

ICI

BP 748,507

Tetrazotised benzidine-3:3'-dicarboxylic acid, 3:3'-dicarboxymethoxybenzidine (I), or 4:4'-diaminoazobenzene-3:3'-dicarboxylic acid is coupled with hydroxyindazoles (whose preparation is described in BP 697,977) to give red to blue direct dyes—



(A = direct link or azo link; B = direct link or  $\text{OCH}_2$ , provided that A be not an azo link if B =  $\text{OCH}_2$ ) which may be aftercouppered. Thus I is tetrazotised and coupled with 6-hydroxyindazole dissolved in aq.  $\text{NaOH}$  and  $\beta$ -ethoxyethanol. The product dyes cotton bordeaux converted to violet on aftercouppered.

E.S.



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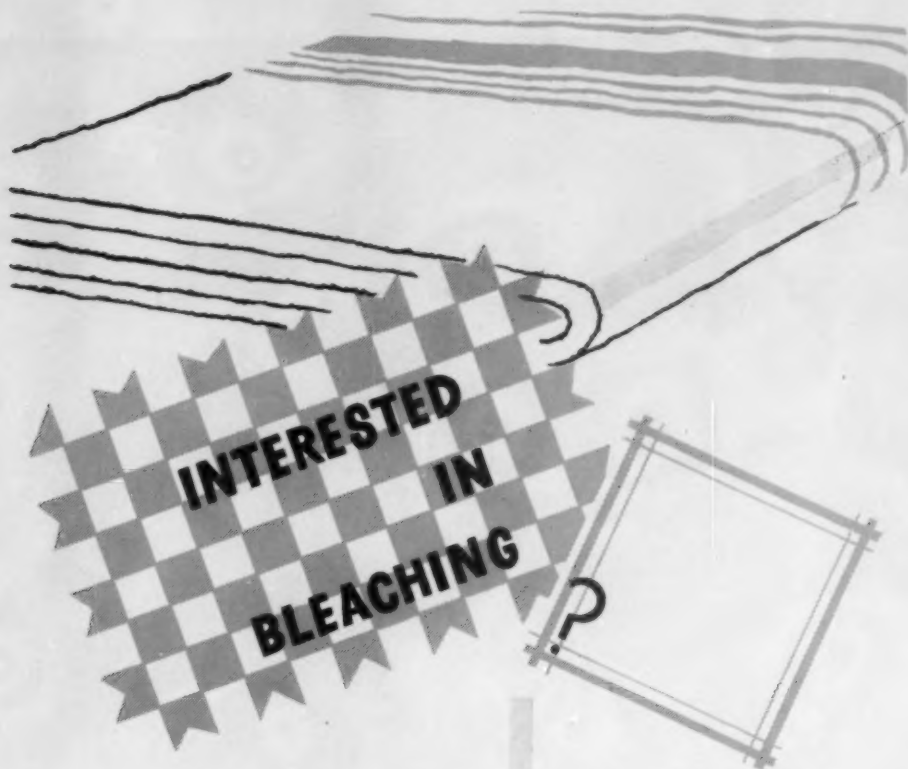
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A. 38

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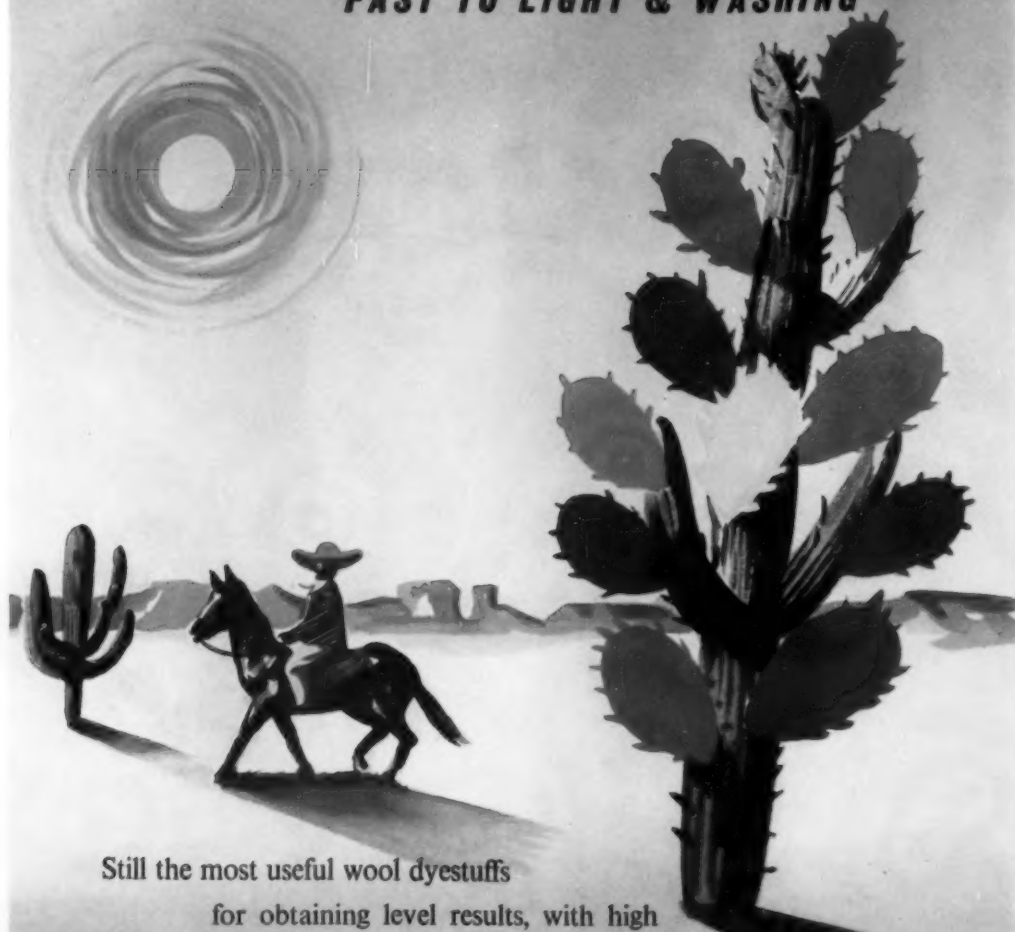
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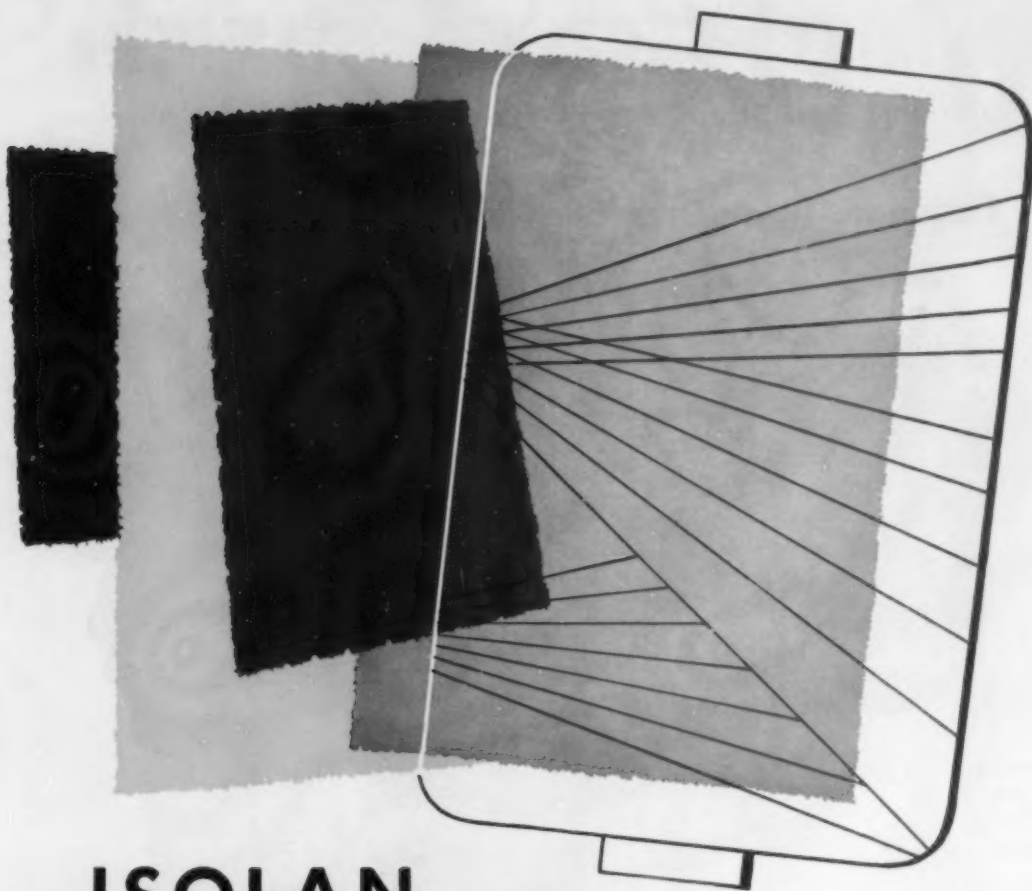
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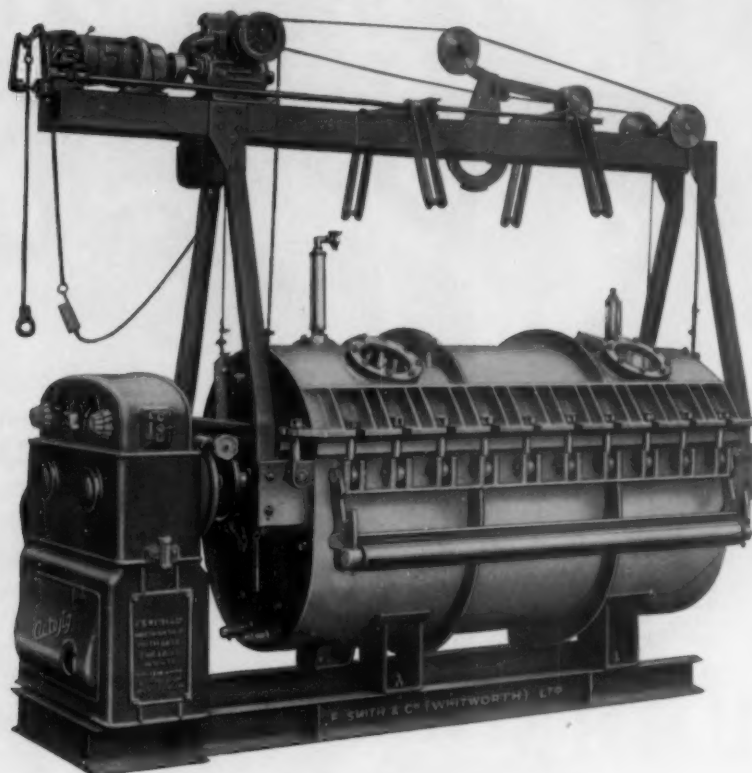
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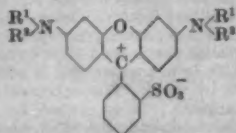


**Triphenylmethane Dyes**

FH

BP 744,972

Red to blue acid dyes fast to light and washing of formula—



are made by condensing the corresponding dihalogeno or dialkoxo compound with a primary or secondary amine  $\text{NHR}^1\text{R}^2$  ( $\text{R}^1 = \text{H}$ , Alk or Ar; and  $\text{R}^2 = \text{Alk}$  or Ar; or  $\text{R}^1$  and  $\text{R}^2$  may together form a cyclic system), and where desirable further sulphonating the product. Thus the dichloro compound, derived by heating with  $\text{POCl}_3$  the phthaloin obtained from *o*-sulphobenzoic acid and resorcinol, is heated with aniline at  $100^\circ\text{C}$ . After removal of excess of aniline the product is sulphonated with  $\text{H}_2\text{SO}_4$ . BP 749,308

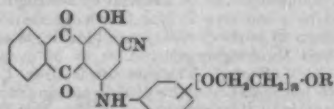
Related asymmetrical derivatives are prepared by treating the dihalogeno compound in turn with different amines, one of which may be chosen to contain OH and COOH groups *ortho* to one another, thus enabling the resulting dye to be e.g. afterchromed. R.K.F.

**Blue Substituted 1-Anilino-3-cyano-4-hydroxy-anthraquinone Disperse Dyes**

Eastman Kodak Co.

USP 2,714,598

Compounds of formula—



( $\text{R} = \text{H}$ ,  $\text{CH}_3$  or  $\text{C}_2\text{H}_5$ ;  $n = 3$  or  $4$ ;  $[\text{OCH}_2\text{CH}_2]_n\text{OR}$  is *o*- or *p*- to the amino group) are blue disperse dyes of good fastness to light and gas-fume fading. Thus the dye 1-hydroxy-2-cyano-4-*p*-( $\beta$ -hydroxy- $\beta$ -ethoxy- $\beta$ -ethoxyethoxy)anilinoanthraquinone is obtained by heating 1-hydroxy-2-bromo-4-*p*-( $\beta$ -hydroxy- $\beta$ -ethoxy- $\beta$ -ethoxyethoxy)anilinoanthraquinone (80 g.) and cuprous cyanide (15 g.) with dry quinoline (640 c.c.) at  $200^\circ\text{C}$ . until no further colour change occurs. This is cooled to  $40^\circ\text{C}$ ., poured into 7% aq. HCl (2.5 litres), heated at  $66^\circ\text{C}$ . for 1 hr. and cooled to  $20^\circ\text{C}$ . overnight. It is then poured into water (9 litres) and stood until the dye settles out. The water is decanted off, the dye washed with water, filtered, well washed with water and dried at  $45^\circ\text{C}$ . C.O.C.

**Sulphuric Esters of Leuco 1-Aminoanthraquinones**

ICI

BP 749,453

The sulphuric esters of leuco 1-aminoanthraquinones are prepared by treating a metal salt of the corresponding leuco compound with a sulphating agent in presence of an amide derived from a secondary amine. Thus 1-aminoanthraquinone is first stirred for 24 hr. at  $40^\circ\text{C}$ . under  $\text{N}_2$  with Zn dust and Na  $\beta$ -naphthalenesulphonate in dimethylformamide. After cooling to  $0-5^\circ\text{C}$ ., dimethylformamide-sulphur trioxide (containing 45%  $\text{SO}_3$ ) is added and stirring continued for 30 min. R.K.F.

**Sulphuric Esters of Leuco 2-Aminoanthraquinones**

ICI

BP 749,607

Leuco derivatives of primary 2-aminoanthraquinones are prepared by treating the leuco compound, or a metal salt of it, with a sulphating agent in a neutral organic liquid in presence of an amide derived from a secondary amine. Thus 2-amino-3-chloroanthraquinone, methyl-triethylammonium chloride, phenol and Zn dust are stirred in ethylene dichloride for 2 hr. at  $40^\circ\text{C}$ . in an inert atmosphere. After cooling to  $20^\circ\text{C}$ ., cyclohexylamine is added and the whole further cooled to  $0^\circ\text{C}$ . while dimethylformamide-sulphur trioxide (containing 45% combined  $\text{SO}_3$ ) is introduced. After 30 min. the leuco sulphuric ester is separated as a solution by pouring into aq.  $\text{Na}_2\text{CO}_3$ , distilling off the solvent, filtering off the Zn residue and concentrating under vacuum. R.K.F.

**Anthraquinone Acid Dyes**

FBY

BP 749,068

Blue neutral-dyeing acid dyes are prepared by condensing 1-amino-4-bromoanthraquinone-2-sulphonic acid with

a hydroaromatic diamine containing no olefinic double bonds and having  $< 1 \text{ NH}_2$  in a hydrogenated nucleus, so that only one  $\text{NH}_2$  reacts. Thus sodium 1-amino-4-bromoanthraquinone-2-sulphonate, hexahydro-*p*-phenylene-diamine, NaOH and  $\text{Cu}_2\text{Cl}_2$  are stirred in water at  $50-70^\circ\text{C}$ ., and the dye is separated by acidifying. The products are also either further sulphonated, acylated or alkylated with an agent which is substituted with  $\text{SO}_3\text{H}$ , NCS, or a heterocyclic compound containing readily replaceable Hal, e.g. cyanuric chloride. R.K.F.

**Anilinohydroxynitroanthraquinones—Disperse Dyes**

Celanese Corp. of America

BP 749,114

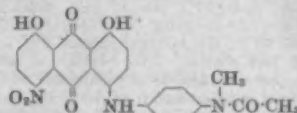
*aa'*-Dinitroanthraquinones containing at least one OH *para* to an  $\text{NO}_2$  group, e.g. 4:8-dinitroanthraquinone, alone or in admixture with 4:5-dinitrochrysin, are condensed in an organic solvent in an inert atmosphere, e.g.  $\text{N}_2$ , with a primary amine, e.g. *p*-aminophenylethanol, to give blue gas-fume-fast disperse dyes. R.K.F.

**Anilinohydroxynitroanthraquinones—Disperse Dyes**

General Aniline

BP 749,036

4:8-Dinitroanthraquinone or 4:5-dinitrochrysin is condensed with amino-*N*-alkyl (or cyanoalkyl)acetanilides to give blue, gas-fume- and light-fast disperse dyes. Thus 4:5-dinitrochrysin and *p*-amino-*N*-methylacetanilide are refluxed for 80 min. in nitrobenzene to produce the dye—



R.K.F.

**Acylaminoanthraquinones—Vat Dyes**

BASF

BP 748,233

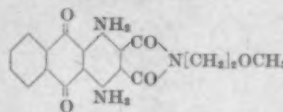
A dialkylbenzene-*m*(or *p*)-dicarboxylic halide is condensed with a vatable amine, or two different aromatic amines one at least of which is vatable, to produce yellow to red vat dyes. The amine may contain (*ortho* to the  $\text{NH}_2$ ) SH, OH,  $\text{NH}_2$  or Hal, in which case the corresponding azole is formed either simultaneously or in a later operation. Thus 2:5-dimethoxyterephthalic dichloride is heated with 1-aminoanthraquinone in nitrobenzene at  $130^\circ\text{C}$ . for several hours. R.K.F.

**1:4-Diaminoanthraquinone-2:3-dicarboximides—Disperse Dyes for Polyester Fibres**

DuP

BP 747,843

Alkoxyalkylamines  $\text{RO}(\text{CH}_2)_n\text{NH}_2$  ( $\text{R} = \text{Alk}$  or Alkoxy of 1-4 C;  $n = 1-4$ ) are condensed with the 1:4-diaminoanthraquinone-2:3-dicarboximide of BP 723,079 (J.S.D.C., 71, 196 (1955)) to give blue disperse dyes suitable for dyeing polyester fibres. Thus the dye—



is prepared by heating for 16 hr. at  $175^\circ\text{C}$ . under pressure 1:4-diaminoanthraquinone-2:3-dicarboximide, aq. 2-methoxyethylamine, methanol and *o*-dichlorobenzene. R.K.F.

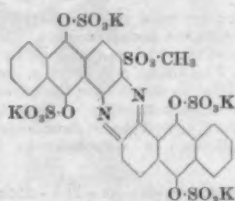
**Bright Greenish-blue Vat Dyes—Sulphuric Half Esters of Indanthrones**

American Cyanamid Co.

USP 2,712,017

A 2-acylamino-3-bromoanthraquinone is converted into a 2-acylamino-3-alkylthioanthraquinone, and then oxidised to a 2-acylamino-3-anthraquinonyl alkyl sulphone. This is then converted into the sulphuric half ester of the corresponding leuco-quinone of the acyl group, hydrolysed, and finally the compound cyclised to produce the indanthrone ring. The products are bright greenish-blue vat dyes of excellent fastness to light and oxidation. Thus  $\text{Na}_2\text{S}$  (83), 8 (21.2), pyridine (360), and water (36) are stirred at  $75^\circ\text{C}$ . until complete solution is obtained. 2-Acetyl-amino-3-bromoanthraquinone (68.6) is added, the mixture is stirred at  $90^\circ\text{C}$ . until the reaction is complete, and it is drowned in water (2000). The mixture is distilled until the distillate reaches  $100^\circ\text{C}$ ., when it is treated with NaCl (120) and cooled to  $20^\circ\text{C}$ . The Na mercaptide is

filtered off, washed with 20% brine (4000), and then slurried in water (2000).  $\text{Na}_2\text{CO}_3$  (80) is added, and dimethyl sulphate (72) gradually stirred in. The yellow ppt. is filtered, washed, dried, and recrystallised from dichlorobenzene to yield 2-acetylaminio-3-anthraquinonyl methyl sulphide. This sulphide (74) is stirred with glacial acetic acid (700) while  $\text{KMnO}_4$  (100) in hot water (30) is added followed by acetic acid (700), and the mixture boiled until reaction is complete. It is cooled to 70°C., and enough  $\text{NaHSO}_3$  is added to decompose the precipitated  $\text{MnO}_2$ . Acetic anhydride (150) is added, and the mixture boiled and drowned in ice and water (2000). The sulphone is filtered off, washed, and dried. To  $\alpha$ -picoline (248) is gradually added at < 30°C. chlorosulphonic acid (89.6), and the mixture kept at 40°C. with stirring. A finely divided mixture of 2-acetylaminio-3-anthraquinonyl methyl sulphone (obtained as above) (70) and iron (26.8) is added slowly, and the whole stirred at 40°C. until reduction of the quinone group and sulphonation are effected. It is then drowned in  $\text{Na}_2\text{CO}_3$  (191.5) in water (1150). The  $\alpha$ -picoline is vacuum-distilled off and the precipitate filtered off and washed with hot water. The filtrate is salted to 23% KCl concentration by volume and cooled to 20°C. The K salt of a sulphuric half ester precipitates, and is washed with saturated alkaline KCl soln., and then with methanol, and dried under vacuum. This half ester (52) is heated in water (490) and 50% NaOH (44) until hydrolysed. Black nickel dioxide prepared from nickelous chloride (64) is stirred in at 80°C., the mixture filtered hot, and the cake washed with hot water. The filtrate is concentrated, preferably under vacuum, to a volume of 400 parts, and KCl added to a concentration of 15% by volume. The mixture is cooled, and the ppt. filtered off and washed with saturated KCl. It is a bright greenish-blue vat dye of excellent fastness, especially to light, and is of formula—



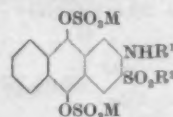
C.O.C.

#### Soluble Vat Dye Intermediates for Greenish-blue Vat Dyes

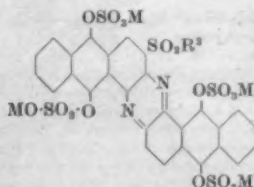
American Cyanamid Co.

USP 2,716,653

Compounds of formula—



( $\text{R}^1 = \text{H}$  or acyl;  $\text{R}^2 = \text{Alk of } < 5 \text{ C}$ ;  $\text{M} = \text{cation}$ ) are intermediates for the leuco sulphuric half esters of bright greenish-blue vat dyes, of good fastness to light and oxidation, and of formula—



Thus  $\text{Na}_2\text{S}$  (83), S (21.2), pyridine (360) and water (36) are heated until complete solution is obtained, when 2-acetylaminio-3-bromoanthraquinone (68.6) is added. When reaction is completed the mixture is drowned in water (2000) and the pyridine distilled off. The hot solution is treated with NaCl (120), cooled to 20°C., the Na mercaptide filtered off, washed with 20% brine (400) and

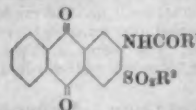
reslurried with water (200).  $\text{Na}_2\text{CO}_3$  (80) is added and the slurry stirred at room temperature while dimethyl sulphate (72 by vol.) is gradually added. The mixture is stirred overnight and the precipitate filtered off, washed neutral, dried and recrystallised from dichlorobenzene to give the yellow 2-acetylaminio-3-anthraquinonyl methyl sulphide. This (74) and glacial acetic acid (700 by vol.) are stirred while  $\text{KMnO}_4$  (100) in hot water (30) is added followed by acetic acid (700 by vol.). The mixture is boiled until oxidation is complete. It is cooled to 70°C., enough  $\text{NaHSO}_3$  added to decompose the  $\text{MnO}_2$  and then drowned in ice-water (2000). The 2-acetylaminio-3-anthraquinonyl methyl sulphone is filtered off, washed neutral and dried. Chlorosulphonic acid (89.6) is gradually added to  $\alpha$ -picoline (248) at < 30°C. and the mixture then brought to 40°C. A mixture of the above sulphone (70) and iron powder (26.8) is gradually added and the mixture stirred at 40°C. until reduction and esterification are complete. It is then drowned in water (1150 by vol.) containing  $\text{Na}_2\text{CO}_3$  (191.5) and the  $\alpha$ -picoline distilled off. The mixture is filtered and the precipitate washed with hot water to a clear wash. The filtrate is salted to 23% KCl by vol. and cooled to 20°C. The K salt of the sulphuric ester of leuco 2-acetylaminio-3-anthraquinonyl methyl sulphone is filtered off, washed with saturated alkaline KCl, then with methanol and dried under vacuum. It is readily soluble in water to give a bright yellow solution. 52 parts of this esterified sulphone are heated gradually to 75°C. with water (490 by vol.) and 50% NaOH (44 by vol.) and kept at 75–80°C. until hydrolysis of the acetylaminio group is complete. The resulting free amino compound can be isolated by salting out or can be oxidised to a vat dye in the following manner:  $\text{NiO}_2$  (prepared from 64 parts of nickelous chloride) is added as a wet cake and thoroughly stirred in at 80°C. until ring closure has been effected. The mixture is filtered, the cake washed with hot water and the filtrate concentrated under reduced pressure to 400 parts by vol. KCl to bring to a 15% concn. by vol. is added and the mixture cooled. The K salt of the tetra-ester of the indanthrone precipitates and is filtered off and washed with saturated aq. KCl. It dyes cellulose a bright blue-green of excellent fastness to light, C.O.C.

#### 2-Acylaminio-3-alkylsulphonylanthraquinones

American Cyanamid Co.

USP 2,716,654

Compounds of formula—



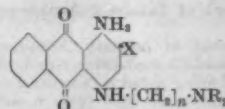
( $\text{R}^1\text{CO} = \text{acyl}$ ;  $\text{R}^2 = \text{Alk of } < 5 \text{ C}$ ), useful as intermediates for vat dyes (cf. USP 2,716,653 (above)), are obtained by heating a suitable halogenoanthraquinone with metallic polysulphides. C.O.C.

#### Blue Anthraquinone Vat Dyes suitable for dyeing Polyacrylic Fibres

DuP

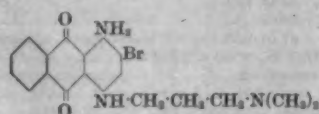
USP 2,716,655

Dyes of formula—



( $\text{X} = \text{Br}$  or  $\text{Cl}$ ;  $\text{R} = \text{Alk of } < 5 \text{ C}$ ;  $n = 2$  or  $3$ ) and their alkylammonium salts are prepared by condensing a 1-amino-2,4-dihalogenoanthraquinone with a dialkylamino-alkylamine. They have good affinity for polyacrylic fibres, being particularly useful as components for greens. Thus 1-amino-2,4-dibromoanthraquinone (25) is heated in pyridine (100) until dissolved. Copper bronze (0.25), 3-dimethylaminopropylamine (25) and K acetate (7.5) are added, and the mixture is stirred under reflux for 3–8 hr. It is cooled, and diluted with water (1500). The precipitate is filtered off, stirred in water (500) and glacial acetic acid (50), heated to 70°C. and filtered. The filtrate is cooled to 15–20°C., rendered slightly alkaline with NaOH, and the

precipitate filtered off, washed and dried. It is a blue dye of formula—



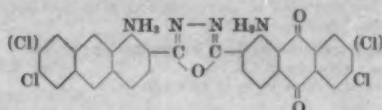
C.O.C.

**Bis-1-amino-6 (and/or 7)-chloroanthraquinonyloxadiazoles—Vat Dyes**

FBy

BP 746,379

Red light-fast vat dyes are prepared by first condensing 1-amino-6 (and/or 7)-chloroanthraquinone-2-carboxylic hydrazide with 1-amino-6 (and/or 7)-chloroanthraquinone-2-carboxylic chloride, e.g. in *o*-dichlorobenzene at 85°C., and cyclising the resulting hydrazide to the corresponding oxadiazole—



by treating with 20% oleum at 25–30°C.

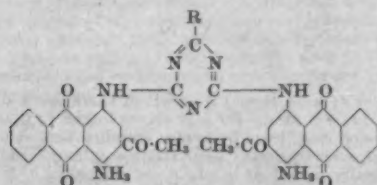
R.K.F.

**Bis(aminoanthraquinonylamino)triazines—Vat Dyes**

BASF

BP 747,454

Blue vat dyes—



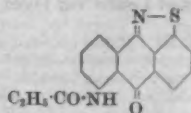
(R = phenyl, which may contain Cl, CH<sub>3</sub> or OCH<sub>3</sub>) are made by heating e.g. 2-phenyl-4:6-dichloro-1:3:5-triazine (1 mol.) with 2-acetyl-1:4-diaminoanthraquinone at 120–210°C. in an inert solvent, e.g. nitrobenzene. R.K.F.

**Acy aminoanthrones—Disperse Dyes**

Ciba

BP 745,086

Yellow to orange disperse dyes are prepared by acylating (aliphatic) a 5-aminoanthrone having a hetero-ring fused at 1:9. Thus 5-amino-1:9-isothiazole-anthrone is refluxed for 30 min. with propionyl chloride in chlorobenzene containing dimethylaniline, to produce the dye—



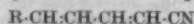
R.K.F.

**Dibenzanthrone Vat Dyes**

Eastman Kodak Co.

USP 2,716,124

Dibenzanthrones containing a cyano group in the 16-position are blue vat dyes which yield dyeings which do not "water-spot", which are exceptionally fast to light and washing, and whose hue is between those obtained with dibenzanthrone and indanthrone. They are made by condensing a 9-methyleneanthrone unsubstituted in at least one of the 1- and 8-positions with a compound of formula—



(R = H, CH<sub>3</sub>, or CN). Thus to 9-methyleneanthrone (20) in nitrobenzene (40) was added, with stirring and below the surface of the liquid at 150°C., 1-cyanobuta-1:3-diene (4) in 15 min. The mixture was then heated at 180–185°C. for 2 hr., cooled to < 100°C., methyl alcohol (25) added, and the mixture chilled to 10°C. The precipitate was filtered off, washed with methyl alcohol, and dried at 110°C. to give a yield of 2-cyano-3:3'-dibenzanthronyl (8–15).

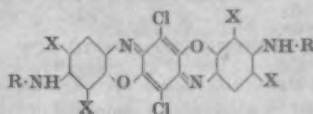
C.O.C.

**Dioxazine Direct Dyes**

Ciba

BP 745,072

Reddish-blue light-fast dioxazine dyes—



(R = *cyclo*Alk; one X = SO<sub>3</sub>H and the other X = Hal), suitable for dyeing and printing animal and vegetable fibres, are made by cyclising in oleum the corresponding quinones, itself prepared by condensing chloranil (1 mol.) with a 4-*cyclo*alkylamino-3(or 5)-halogenoaniline-6(or 8)-sulphonic acid in water containing an acid-binding agent. R.K.F.

**Fluorescent Brightening Agents**

FBy

BP 748,884

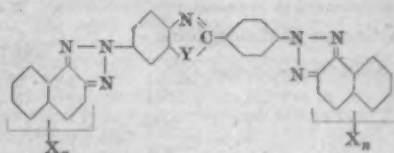
Fluorescent brightening agents, readily soluble in water and suitable for animal, vegetable or synthetic fibres, are obtained by treating a difficultly water-soluble fluorescent amine with an aliphatic sulfone of > 2 C. Thus 2-*p*-aminophenyl-5-methylbenzothiazole (5) is mixed with butane sulfone (6) and melted at 140–150°C. The temperature of the clear melt quickly rises to 164°C. with outer heating. The melt then solidifies to yield a product which when powdered is a light yellow and dissolves easily in warm water. It shows blue fluorescence. C.O.C.

**Fluorescent Brightening Agents**

DuP

USP 2,715,630

Compounds of formula—



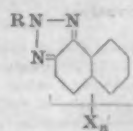
(X = sulpho; n = 1 or 2; Y = O, S, or NH), obtained e.g. by coupling tetrazotised 6-amino-2-*p*-aminophenylbenzoxazole with two molecules of sodium naphthionate, have bluish fluorescence, are stable to hypochlorites, have good affinity for cellulosic fibres, on which they show good build-up, and so are suitable for use in household detergents. C.O.C.

**Fluorescent Brightening Agents for Cellulosic Fibres**

DuP

USP 2,715,632

Compounds of formula—



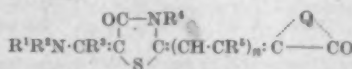
(R = *p*-radical of 2-phenylbenzoxazole, 2-phenyl-methylbenzoxazole, 2-phenyl-methoxybenzoxazole, or of their sulpho derivatives; X = water-solubilising acid radical; n = 1 or 2) may be produced by diazotising the corresponding 2-*p*-aminophenylbenzoxazole and coupling in acid medium with 1- or 2-aminonaphthalene having one or two sulpho groups or a carboxy group in the nucleus, the resulting *o*-aminoazo dye being then oxidised to a triazole, i.e. it is oxidised until it becomes colourless. They have strong blue fluorescence, are stable to hypochlorite, and are suitable for use in detergent compositions. C.O.C.

**merocyanine Dyes containing an Aminomethenyl Group**

Eastman Kodak Co.

USP 2,713,579

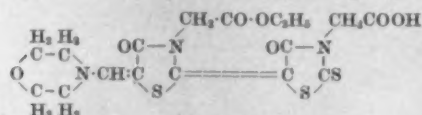
Dyes of formula—



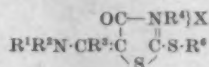
(R<sup>1</sup> and R<sup>2</sup> = H, Alk, acyl, Ar, *cyclo*alkyl or together = atoms to complete a heterocyclic ring; R<sup>3</sup> = H or Alk;



$R^4 = \text{Alk}$ ;  $R^5 = \text{H}$ , Alk or alkoxy;  $n = 0$  or 1;  $Q = \text{atoms}$  to complete a 5-6-membered heterocyclic ring, e.g.—



are obtained by condensing a compound of formula—



( $R^6 = \text{Alk}$  or Ar;  $X = \text{anion}$ ) with one of formula—



C.O.C.

### Phthalocyanine Dyes

BASF

BP 749,349

A phthalocyanine is treated with chlorosulphonic acid and the resulting polysulphonyl chloride reacted with aq. 4-amino-4'-nitrodiphenylamine-2'-sulphonic acid. Unreacted chlorosulphonyl groups are hydrolysed with alkali, the resulting dye being suitable for colouring leather, paper and varnish lacquers. Thus copper phthalocyanine is heated for 4 hr. at 135-140°C. with chlorosulphonic acid. After separating by pouring into ice and water, the sulphonyl chloride thus formed is stirred at 15-20°C. for 30 min. with an aq. ammoniacal solution of 4-amino-4'-nitrodiphenylamine-2'-sulphonic acid and the resulting paste evaporated to dryness.

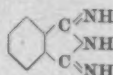
R.K.F.

### Leuco Copper Phthalocyanine

DuP

BP 748,854

A 1:3-diiminoisindoline—



or a 4:5-benzo homologue is heated with a cupric salt in an inert organic solvent, e.g. ethylene glycol monoethyl ether, at 55-95°C. to produce a leuco copper phthalocyanine.

R.K.F.

### Phthalocyanine Pigments

DuP

BP 746,901

Monochloro-copper phthalocyanine is prepared directly in a pigmentary state by heating phthalonitrile and micro-pulverised  $\text{Cu}_2\text{Cl}_2$  in an inert organic liquid, e.g. nitrobenzene, at 190-215°C.

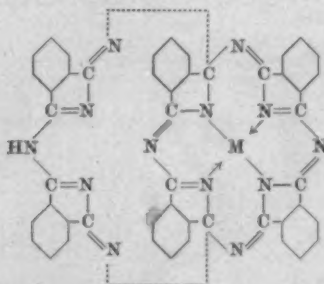
R.K.F.

### Phthalocyanine Derivatives

DuP

BP 745,359

Phthalonitrile, ammonia and an anhyd. Ni or Cu salt are heated together at  $\geq 90^\circ\text{C}$ . in presence of a water-soluble solvent, e.g. glycol monoethyl ether, and a catalyst, e.g. methylglucamine, to produce a yellow-brown phthalocyanine derivative, which may be applied to a textile material from a solution in a solvent, e.g. glycol monoethyl ether. On heating to 120-200°C. or treating with a reducing agent, e.g. K ferrocyanide, and lactic acid the phthalocyanine proper is formed on the fibre, probably by a reaction which comprises loss of 2 mol. of phthalonitrile and 1 mol. of  $\text{NH}_3$  from the triplanar structure—



R.K.F.

### Non-flocculating, Non-crystallising Phthalocyanine Pigments

American Cyanamid Co.

BP 746,704

Phthalocyanine pigments which do not flocculate or crystallise, e.g. in nitrocellulose lacquers, are prepared by treating with  $\text{NH}_3$  or an aliphatic primary amine, a phthalocyanine containing  $\geq 2$  *o*-carboxybenzamidomethyl groups per mol. but  $\leq 1$  per 20 mol. (made as described in BP 695,523—J.S.D.C., 69, 465 (1953)). Thus the product made by condensing copper phthalocyanine with hydroxymethylphthalimide in  $\text{H}_2\text{SO}_4$  is boiled with aq. ethylenediamine.

R.K.F.

### Acid Pasting of Phthalocyanine Pigments

General Aniline

USP 2,716,649

The dry pigment is milled with 2.5 times its wt. of a strong acid, e.g. 80%  $\text{H}_2\text{SO}_4$ , the resulting thick magma drowned in water and the pigment filtered off and washed free of acid. Good results are obtained with a brief milling period, e.g. 5 min., and the process is applicable to pigments which are unstable to conventional acid pasting, e.g. to metal-free phthalocyanines.

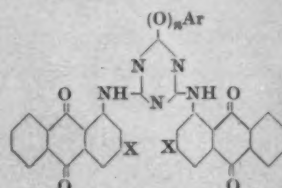
C.O.C.

### Soluble Esters of Yellow Triazine Vat Dyes

American Cyanamid Co.

USP 2,716,645

Esters of yellow dyes of formula—



( $n = 0$  or 1;  $X = \text{H}$  or Cl) in which there are 4 sulphate groups, i.e. 4 S atoms for 5 N atoms linked to C atoms of the triazine ring, have adequate stability and yield deep, fast and even dyeings. Thus chlorosulphonic acid (17.7) is gradually added at 10-15°C. to  $\alpha$ -picoline (130). The mixture is kept under  $\text{N}_2$  while the yellow dye (10) corresponding to the above formula when  $n = 1$ ,  $\text{Ar} = \text{C}_6\text{H}_5$ , and  $X = \text{H}$  is added. Powdered iron (7.2) is then gradually added at 29-34°C. The mixture is then stirred for 3 hr. at 40°C. and finally at room temperature overnight. The mixture is then drowned in water (600) containing  $\text{Na}_2\text{CO}_3$  (55) and the picoline removed by vacuum distillation. The residue is heated, filtered and vacuum-concentrated to the volume of about 330 parts water.  $\text{KCl}$  (82.5) is added at 10°C. to salt out the product, which is filtered off and washed with  $\text{KCl-K}_2\text{CO}_3$  soln. An excellent yield of S:N ratio 0.78 is obtained. The product yields brilliant yellow dyeings.

C.O.C.

### Sulphuric Esters of Leuco Vat Dyes

DH

BP 745,365

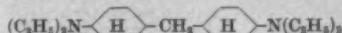
The method of BP 719,621 (J.S.D.C., 71, 167 (1955)), for preparing sulphuric esters of leuco vat dyes which are not readily converted to that form, is improved by using, instead of or in addition to finely divided Cu or brass, at least one of the metals Fe, Ni and Co. Thus 1:5-dibenzoyl-aminoanthraquinone is heated with Fe powder for 5-6 hr. at 55°C. in a mixture of diethylcyclohexylamine and commercial  $\alpha$ -picoline (b.p. 125-128°C.) containing chlorosulphonic acid.

BP 745,368

The processes of the above patent are modified by using, instead of, or in addition to, diethylcyclohexylamine, at least one diethyl-(monoethylcyclohexyl)-amine in a total proportion of 10-60% by weight of the  $\alpha$ -picoline.

BP 745,088

The cyclohexylamine derivatives used in the previous two patents are replaced by methylene-4:4'-bis(NN-diethylcyclohexylamine)—



R.K.F.

### Increased Yields of Tar-free Carbon Black

Phillips Petroleum Co.

USP 2,714,055

In a method in which an axial stream of hydrocarbon gas surrounded by a helically moving blanket of hot gas is



passed axially into a reaction zone, increased yield is obtained by producing larger quantities of tarry pigment than the maximum possible production of tar-free pigment and then detarring the product. C.O.C.

#### Carbon Black

Phillips Petroleum Co.

USP 2,716,053

A carbonaceous material is partly burnt so as to produce a gas containing a large proportion of CO free from elemental C. This hot gas is quenched with a relatively cold tail gas stream at a temperature at which the CO reacts at a high rate to form carbon black but below that at which undesirable side-reactions occur. C.O.C.

#### Red Hydrous Ferric Oxide

C. K. Williams & Co.

USP 2,716,595

Soft-textured brilliant red iron pigments free from deleterious impurities are produced by wet chemical precipitation at < 100°C. by oxidising metallic iron suspended in aq. ferrous sulphate or chloride in presence of O<sub>2</sub>-containing gases and a suspension of a suitable iron compound. The colour of the pigment and its other properties are controlled by the amount of insoluble iron compound employed, temperature, pH and degree of oxidation. C.O.C.

#### Pigment Paste

S. N. Hunter

BP 748,410

A plastic pigment paste which is compatible with the usual paint and ink vehicles and in addition is hydrophobic, is highly heat-resistant, and remains soft and usable indefinitely, comprises a pigment, wetted or coated with at least 5% of its wt. of chlorinated polyphenyl. The chlorinated polyphenyl forms 15-90% by wt. of the pigment paste. The products have a higher degree of brightness, specular gloss and reflectivity than hitherto known pigment pastes. Metal pigments used in this way lose no lustre even on prolonged exposure to air, moisture and corrosive environments. C.O.C.

#### Pigments

Fran

BP 748,670

A basic dye is dispersed in a water-insoluble polyvalent-metal salt of a highly polymerised unsaturated carboxylic acid or a copolymer of such an acid. This gives cheap pigments of great brightness and excellent fastness to water and organic solvents. Thus, polyacrylic acid (500 parts) is neutralised with triethanolamine (160) diluted with water (1000); Rhodamine B extra conc. (7.9) and Rhodamine 6J extra conc. (2) are then added. To the resulting solution is added ZnSO<sub>4</sub> (100) dissolved in water (1000). The precipitate is pressed, washed, dried and ground. The product is a bright red luminescent pigment with high fluorescence and very good fastness to light, water and solvents. C.O.C.

Zinc Dust as a Protective Pigment (V this page)

Electron-microscopical and diffraction Study of Solutions of Benzopurpurin 4B (VIII p. 400)

Colloidal Chemistry of Haematein Salts (VIII p. 400)

Ultraviolet-radiation-absorbing Compounds and their Use in Colour Photography (IX p. 401)

Spectrophotometric Determination of Magnesium with Sodium 1-Azo-2-hydroxy-3-(2:4-dimethylcarbox-anilido)-naphthalene-1'-(2-hydroxybenzene-5-sulphonate) (XIV p. 407)

Coal-tar Colours. XX—FD&C Blue No. 2 (XIV p. 408)

Triphenylmethane Dyes derived from Ethylbenzylaniline-sulphonic Acid (XIV p. 408)

### V—PAINTS; ENAMELS; INKS

#### Effect of Particle Size on the Oil Absorption of Pigments

W. Kaufmann

Deut. Farben-Z., 10, 45-46 (1956); Chem. Abs., 50, 6810 (10 May 1956)

The particle size (in  $\mu$ ) and oil absorption of the following commercial pigments, of their fines (obtained by sedimentation), and of very finely ground pigments are—Chrome Yellow 70, 18.4; 30, 14.9; 10, 24.6; Chrome Green 25, 27.5; 11, 22.5; 3, 23.1; Red Iron Oxide 70, 23.7; 12, 23.8; 6, 24.0; Furnace Black 28, 51.8; 20, 53.8; 10, 59.4; Lithopone 35, 13.8; 24, 13.5; 3, 13.2; Milori Blue 73, 64.4; 10, 63.7; 5, 63.3; Permanent Red 30, 68.3; 15, 59.8; 4,

64.4; Lamp Black 20, 192.0; —, —, 3, 145.2. Treatment in jet mills destroys the porosity of pigments, with consequent lowering of their oil absorption. C.O.C.

#### Stability of Cellulose Bronze Lacquers

W. F. Daggett

Paint Technol., 19, 299-300 (1955); Chem. Abs., 50, 6068 (25 April 1956)

Bronze powders made from Cu and Cu alloys can cause gelation of cellulose nitrate lacquers and tend to turn many vehicles green. Approx. 0.3% of boric, malic, citric or oxalic acid can be added to such lacquers to prevent gelling, but may retard drying and reduce gloss. Use of adding water and H<sub>2</sub>O<sub>2</sub> to obtain stable viscosity is mentioned. The green coloration is caused by the Cu reacting with acids from solvent hydrolysis and/or from resins present. Its formation may be prevented by adding oxalic acid, sodium acetate and substituted ureas. Suitable cellulose acetobutyrate and ethylcellulose lacquers produce non-gelling, non-greening, bronze finishes. C.O.C.

#### Zinc Dust as a Protective Pigment

O. Roder

Farbe und Lack, 62, 51-58 (1956)

#### Lithopone in Emulsion Paints

J. G. Campbell

J. Oil & Colour Chem. Assocn., 39, 212-214 (March 1956)

The author replies to certain criticisms made by J. D. Rigg (*ibid.*, 38, 724 (1955)) concerning his paper on the exterior durability of emulsion paints (*ibid.*, 36, 550 (1955)). It is confirmed that the tests reported related to the exterior durability, and that ZnS paints were undistinguished in performance. Erosion of the ZnS paints was more rapid than suggested by the chalking curves, since checking, crazing, and flaking also occurred in varying amounts; six photomicrographs are reproduced in illustration. The reported green discoloration of ZnS paints did not occur in every test series, but it did not occur at all with other pigments in otherwise identical paints under identical conditions. All ZnS paint exposures developed a grey discoloration; this is probably due to dirt retained on the surface of low pigmentations, and in the cracks of the medium and high ones. J.W.D.

#### PATENTS

#### Magnetic Ink

Burroughs Corp.

BP 748,763

Powdered hydrogen-reduced iron, electrolytic iron, Swedish sponge iron, "carbonyl" iron or other ferromagnetic material of equal or better permeability, no particle being > 5  $\mu$ , is dispersed in a permanently fluid viscous mixture of organic esters, glycols or oleic and lauric acids. The metal forms 50-70% of the final product, which is used for production of magnetic records on paper by printing, typing or the like. C.O.C.

#### Crackle Finish

Sherwin-Williams Co.

USP 2,714,560

Pigment Paste (IV this page)

### VI—FIBRES; YARNS; FABRICS

#### Engineering Aspects of Textile Structures

K. R. Fox

Amer. Dyestuff Rep., 45, 317-324, 346 (21 May 1956)

The physical properties of different fibres and their influence on fabric structure and behaviour are discussed, and a parallel is drawn between these materials and the diverse materials available, for selection according to end-use, in the engineering industry. J.W.B.

#### Chemical Characteristics of Jute and Hibiscus Fibres

W. G. Macmillan, A. B. Sen Gupta, and A. Roy

J. Indian Chem. Soc., 33, 49-53 (Jan. 1956)

A comparison of the chemical composition of jute with that of bast fibres of the *Hibiscus* group—viz. mesta, bimbi, and kenaf—has shown that there is a difference in the contents of lignin (12-14.8%, 9.5-10.8%), acetyl group (3.1-3.8%, 4.5-5.2%), total acidity (100-114, 128-146 m-equiv./100 g.), and methoxy groups associated with the carbohydrate fraction (0.85-0.95%, > 1.25%). C.H.R.

#### Production and Processing of Coconut Fibres

W. Frenzel and H. Neumann

Faserforsch. und Textiltech., 7, 143-152 (April 1956)

### Properties of Solutions of Cellulose Xanthates of Low Degree of Substitution—I

N. I. Nikitin and E. A. Abramova

*J. Appl. Chem. U.S.S.R.*, **29**, 253-260 (Feb. 1956)

Nikitin and coworkers have previously demonstrated the possibility, in principle, of preparing soln. of cellulose xanthate by the use of only 10-15% of  $\text{CS}_2$  on the wt. of alkali-cellulose, the essential feature of the method being a very low dissolution temp. Practical conditions for the preparation of readily filterable weakly xanthated viscose are now investigated, and very stable products of this kind are obtained with as little as 6% of  $\text{CS}_2$ . It is found that the ammonium chloride value is of no use as an index of the ripening of such viscose. Although the viscosity of viscose is greatly reduced by freezing and rethawing, it is found that the D.P. of cellulose regenerated from the soln. is unchanged, and the change in viscosity must be attributed to the breaking up of molecular aggregates in the freezing process.

A.E.S.

### Influence of Cellulose Concentration in the Viscose on the Physical Properties of Viscose Rayon Fibres

H. Klare

*Faserforsch. und Textiltech.*, **7**, 199-207 (May 1956)

Examination of viscose rayon fibres obtained from viscose solutions of increasing cellulose concentration shows that the D.P. is the main factor determining strength, the actual cellulose concn. being of secondary importance. If the cellulose content is increased, degradation cannot be compensated for by extended pre-ripening.

W.R.M.

### Effect of Extension of Cellulose Xanthate Filament

S. Mukoyama and Y. Tsuda

*J. Soc. Textile Cellulose Ind. Japan*, **12**, 188-192 (March 1956)

Isotropic cellulose xanthate filament shrinks on regeneration and drying, while the stretched filament shows diminished shrinkage under the same conditions. The degree of swelling of xanthate filament decreases with the degree of extension. A similar relationship holds for the freshly regenerated cellulose filament which has been pre-extended in the xanthate state. The anisotropy of swelling of xanthate filament increases with extension, and the filament shows almost the same value after regeneration. These effects are more marked with  $\text{NH}_4$  xanthate than with  $\text{Zn}$  xanthate. The swelling of cellulose filament regenerated from  $\text{Zn}$  xanthate is lower than that from  $\text{NH}_4$  xanthate.

AUTHORS

### Change in Fibre Structure of Regenerated Cellulose Fibres on Treatment with Caustic Soda Solutions

T. Yurugi

*J. Soc. Textile Cellulose Ind. Japan*, **12**, 96-99 (Feb. 1956)

Changes in fibre structure of regenerated cellulose fibres, such as degree of crystallinity and lateral order and cross-sectional shape, occurring on treatment with aqueous alkali of various concentrations, have been investigated experimentally. Rayons of two types were used and compared: Fibre S, ordinary viscose rayon of irregular section and non-uniform distribution of lateral order; and Fibre X, a special viscose rayon having uniform round section and uniform single-peak lateral-order distribution. Both fibres showed increases in crystallinity after swelling and deswelling, but the magnitude of the change in the low-ordered fibre (S) was much greater. The skin effect of this fibre disappeared, and the distribution of lateral order shifted to a highly ordered level and showed a similar curve to that of Fibre X. Changes in the physical properties of fibres produced by swelling treatments were also observed, and are discussed in terms of the relation between fibre structure and mechanical properties. It is concluded that the physical properties of Fibre X are closely associated with crystallinity and orientation, whereas those of Fibre S are much more markedly affected by the skin-core structure.

AUTHOR

### Performance Characteristics of Synthetic Fibres, Wool, Viscose Rayon, and Blended Fibres in Axminster Carpets

J. W. Schappel

*Text. Research J.*, **26**, 211-226 (March 1956)

Nylon, Dacron, Orlon, saran, dynel, cellulose acetate, wool, and smooth viscose rayon, as well as 50:50 blends of the synthetics and wool with smooth viscose rayon, were processed (in natural colour) into standard seven-row

Axminster fabric. Samples were tested under service conditions for resistance to soiling and crushing. In addition, the fibres were examined in staple form for soil pick-up and retention characteristics; clean fibres and soiled fibres from service-tested samples were examined by optical and electron-microscopical methods; and elastic recovery and moisture regain, properties related to crush resistance, were investigated. Soiling properties are influenced by the smoothness, available surface area, and reflective properties of the fibre. Static electricity has little influence on soiling. Smooth viscose rayon has the highest soil resistance of all the fibres tested, followed by wool and cellulose acetate. The synthetic fibres are markedly more susceptible to soiling. The soiling properties of fibres in blends are not influenced by other types of fibre present. Crush-recovery characteristics of the synthetic fibres are good, nylon and Orlon being superior to wool. Blends of the synthetics and smooth viscose rayon show poorer crush recovery than the average crush properties of the respective fibres. Differences in pile density of the fabrics as a result of differences in fibre density are not sufficiently great to affect the crush-resistance ranking of the fibres.

J.C.F.

### Amino-acid Composition of Keratins

D. H. Simmonds

#### II—Amino-acid Composition of a Keratin Derivative extracted from Wool with Alkaline Thioglycollate Solution

*Australian J. Biol. Sci.*, **8**, 114-121 (Feb. 1955)

The amino-acid composition of a protein component of 64s merino quality wool, which moves as a single peak on electrophoresis in alkaline thioglycollate solutions, has been determined. The results show that the purified protein fraction contains more aspartic acid, glutamic acid, leucine, lysine, and amide nitrogen, and less cystine, proline, serine, and tryptophan, than the parent wool from which it was extracted.

#### III—Amino-acid Composition of Different Qualities of Wool

*Ibid.*, **8**, 537-540 (Nov. 1955)

The amino-acid composition of acid hydrolysates of three commercially classified wools has been determined. The overall pattern of the amino-acid composition of the three wools is similar, although small variations between the wools are observed with some of the amino acids.

P.G.M.

### Bilateral Structure of Wool Cortex and its Relation to Crimp

R. D. B. Fraser and G. E. Rogers

*Australian J. Biol. Sci.*, **8**, 288-299 (May 1955)

New evidence of the nature of the bilateral cortex in crimped wool is presented and discussed in relation to the physical and chemical properties of the wool fibre. The origin of the asymmetry in the cortex was studied by cutting cross-sections of sheepskin and swelling plucked wool roots. It is shown that the bilateral structure is present before the cortex is keratinised and does not originate from an asymmetrical keratinisation, as has been suggested. Coarse wools, in which crimp is much less, are shown to possess a radial asymmetry, in which the peripheral cortical cells are less accessible to basic dyes and have a higher sulphur content than the central cells. This is related to the difference between the curved follicles, from which fine fibres are produced, and the straighter follicles, producing coarse fibres.

P.G.M.

### Dimensional Changes and Related Phenomena in Wool Fibres under Stress

E. C. Banky and S. B. Slen

*Text. Research J.*, **26**, 204-210 (March 1956)

Transverse and longitudinal measurements were made on wool fibres at different stages of elongation up to the breaking point. Decrimping was found to be associated with a slight but significant decrease (1.2% at  $P < 0.01$ ) in fibre diameter. The longitudinal contraction due to crimp was calculated to be 2.3% of the decrimped fibre length. The overall rate of decrease in fibre diameter as related to the rate of extension indicated a constant volume distortion beyond the Hookean extension in stretched fibres. Slight but significant ( $P < 0.05$ ) deviations from the general trend at about 5% and 27% extension suggest the possibility of drastic structural changes at these points. Poisson's ratio was found to be 0.63 in the region of

Hookean extension, whereas in the later stages of extension it approached 0.46. Measurement of the diameter of the broken ends of fibres 24 hr. after rupture showed that the average cross-sectional area at the point of rupture was 32% smaller than the average for the entire fibre. This is taken as indicating that, for undamaged fibres, maximum extension resulting in rupture occurs at the thinnest segment. None of the stretched fibres showed visible mechanical damage to any part of the structure.

J.C.F.

#### Reaction of Wool Keratin with Alkali

S. Blackburn and G. R. Lee

*Biochim. Biophys. Acta*, **19**, 505-512 (March 1956)

Amino-acid analyses have been made on normal wool and wool treated with boiling alkaline buffers: the only amino acid to undergo appreciable destruction is cystine, serine and threonine being destroyed to a small extent. Determination of lanthionine in a number of wools treated with alkali under differing conditions shows that for every cystine residue attacked during the alkaline treatment one residue of lanthionine is produced. During the degradation of cystine, therefore, reactions other than lanthionine formation do not take place to any appreciable extent.

P.G.M.

#### Rabbit Fibres. V—Geometry of Scale Forms of Some Animal Fibres

S. Ikeda and S. Okajima

*J. Soc. Textile Cellulose Ind. Japan*, **12**, 198-202 (March 1956)

#### Infrared Studies of Polypeptides related to Silk

A. Elliott and B. R. Malcolm

*Trans. Faraday Soc.*, **52**, 528-536 (April 1956)

Infrared and X-ray powder studies of regenerated, water-soluble forms of silk spun by *Anaphe moloneyi* show the presence of  $\alpha$ -helical configurations when the regenerating agent is trifluoroacetic acid. When the water-soluble material is freeze-dried from aqueous solutions, the characteristic diffraction pattern of the  $\alpha$ -helix is not found. Spectra of the oriented  $\beta$ -form of the silk have been observed. Infrared spectra and X-ray photographs of two molecular configurations of polyglycine have been obtained, and their significance is discussed.

W.R.M.

#### Infrared Spectra and Structure of the Proteins of the Silk Glands

H. Lenormant

*Trans. Faraday Soc.*, **52**, 549-554 (April 1956)

The infrared spectra of water-soluble fibroin and the proteins of the silk glands are very similar, but silk-gland proteins can be distinguished from fibroin by a group of quite prominent bands from 1200 to 950  $\text{cm}^{-1}$ . Silk-gland proteins become insoluble after crushing and stretching, and a film has a spectrum that shows the characteristics of the  $\beta$ -structure. The formation of the insoluble silk thread is not a simple crystallisation but a real denaturation with structural change. The study of deuterated products in the dry state and in heavy-water solution shows a slight shift of the 1450  $\text{cm}^{-1}$  band. This does not occur with soluble proteins which have  $\alpha$ -type structure, and it is likely that the structure of the water-soluble fibroin and the silk-gland protein show some peculiar features connected with their sensitivity to mechanical denaturing factors.

W.R.M.

#### Crystal Structure of Silk Fibroins

J. O. Warwicker

*Trans. Faraday Soc.*, **52**, 554-557 (April 1956)

An X-ray examination of nine different silk fibroins shows that they can be divided into three groups, typified by the fibroins of *Bombyx mori*, *Antheraea mylitta*, and *Anaphe moloneyi*. The X-ray diagrams can be explained on the basis of orthorhombic unit cells, two of the cell dimensions being common to the three types. The cell dimension that varies from type to type is thought to be in the direction of the side-chains of the polypeptide molecules.

W.R.M.

#### X-Ray Investigation of Silk Fibroin

O. Kratky

*Trans. Faraday Soc.*, **52**, 558-570 (April 1956)

Investigations on silk as a native gel, as renatured solution, and in various forms of the solid state are reported. Transformations of the protein are discussed.

W.R.M.

#### Interaction of Nylon with Aqueous Solutions of Hydrochloric Acid

F. T. Wall and A. Beresiewicz

*J. Phys. Chem.*, **60**, 692-698 (May 1956)

The adsorption of HCl by nylon is studied as a function of acid concentration. The initial stages of absorption follow theoretical expectations based on a model involving interaction of HCl with polymer end-groups. If the nylon has excess of amino over carboxyl end-groups, absorption occurs in two stages, which can be described thermodynamically. In conc. HCl nylon absorbs more HCl than is to be expected from the number of amino groups. This excess absorption is attributed to interaction with amide linkages. Heats of absorption are calculated for each of the stages.

W.R.M.

#### Alkaline Polymerisation of 6-Hexanolactam (Rapid Polymerisation)

W. Griehl

*Faserforsch. und Textiltech.*, **7**, 207-212 (May 1956)

The course of the rapid alkaline polymerisation of  $\epsilon$ -caprolactam is studied, using various initiators. The changes of D.P. and mol.wt. distribution with time are examined. End-group determinations are made, and a new mechanism of alkaline polymerisation is suggested in which metallo-organic binding is hydrolytically broken by contact with water and the lactam ring is broken with formation of a carboxyl group. On remelting, carboxyl and amino groups react to produce longer chains.

W.R.M.

#### Hydrogen Bonding in 6-Hexanolactam and 6-Nylon

V. N. Nikitin

*Izvestiya Akad. Nauk S.S.S.R., Otdel. khim. nauk*, **92-97** (Jan. 1956)

The infrared absorption spectra of 6-hexanolactam, *N*-deutero-6-hexanolactam, and 6-nylon are determined over a wide range of temp. The results do not support the view (see J.S.D.C., **69**, 227 (1953); **70**, 477 (1954); **71**, 120 (1955)) that the different maxima in the NH band are to be attributed to hydrogen bonds of different types, and the view that there are three types of hydrogen bonds in 6-nylon must be regarded as doubtful.

A.E.S.

#### Process of Formation of Polyamide Resins. V—Rôle of Polymerisation and Polycondensation Reactions in the Process of Converting 6-Hexanolactam into Polymer

A. S. Shpil'n'yi

*J. Gen. Chem. U.S.S.R.*, **26**, 530-534 (Feb. 1956)

Previous work by the author indicates that, in presence of water, 6-nylon is formed from 6-hexanolactam both by stepwise polymerisation and by polycondensation. In confirmation of this view, the effect of water concn. on reaction rate and on amounts and mol.wt. of macromolecular and water-sol. products is determined, and attempts are made to isolate the component processes—first, polycondensation is excluded by carrying out reaction in complete absence of water, adipic acid being used as catalyst, and polymerisation is then excluded by working, in complete absence of 6-hexanolactam and water-sol. substances, with intermediate products (mol.wt. = 3000) in the formation of 6-nylon. The rate of polycondensation is found to be much greater than that of stepwise polymerisation. Throughout the whole process the polymer fractions of low mol.wt. form an insignificant part of the mixture, this fact being explained by the rapidity of polycondensation as compared with lactam hydrolysis. The rate of the process is greatly dependent on the total number of functional groups present, and the great accelerating effect of water is due to increase in the number of functional groups, which favours both polymerisation and polycondensation.

A.E.S.

#### Cyclic Oligomers of 6-Hexanolactam

O. Fukumoto

*Bull. Chem. Soc. Japan*, **29**, 169-171 (Jan. 1956)

From the hot-water extract of 6-nylon two cyclic dimers, one of which may be a diketopiperazine, and one trimer are obtained.

A.J.

#### Cold Drawing of High Polymers—The Quasi-static Drawing of Polycapramide. I—Temperature Dependence, Softening Temperature, and Second-order Transition Point. II—Effects of Humidity

H. Yumoto

*Bull. Chem. Soc. Japan*, **29**, 45-51 and

141-147 (Jan. 1956)



### 1:8-Diazacyclotetradecane and 1:8:15:22-Tetraazacyclooctacosane [from Nylon]

H. Zahn and H. Spoor

*Chem. Ber.*, **89**, 1296-1302 (May 1956)

The cyclic diamide obtained from the technical, aqueous extracts of polycaprolactam was reduced to the first-named cpd. by  $\text{LiAlH}_4$  in tetrahydrofuran. The other cpd. is prepared similarly, but also starting from aqueous extracts of 6,6-nylon. Derivatives of both substances are made, and evidence for others is presented. H.E.N.

### Recovery from Bending of Wires and Filaments of Polyethylene Glycol Terephthalate

H. Ludewig and G. Glöckner

*Faserforsch. und Textiltech.*, **7**, 221-226 (May 1956)

In spite of the crease resistance of polyethylene glycol terephthalate textiles, bristles of the material do not rise after being pressed down. Measurements of crease angle and density of wires and filaments show that bodies formed with a large diameter are easily deformed permanently by bending. The power of crease resistance of polyethylene glycol terephthalate increases with crystallinity and may be improved by heat-treatment. W.R.M.

### Acrylic Fibres

A. Hunyar and H. Reichert

#### XIII—Solubility of Polyacrylonitrile

*Faserforsch. und Textiltech.*, **7**, 165-170 (April 1956)

Increased temperature of polymerisation caused decreased solubility in dimethylformamide. Solubility can be decreased by thermal treatment in the aqueous phase. This aftertreatment is not effective with copolymers.

#### XIV—Graft Polymerisation with Polyacrylonitrile Backbone for Fibre Purposes

*Ibid.*, **7**, 213-221 (May 1956)

Graft copolymers are prepared, by chain transfer, of polyacrylonitrile and polyvinyl acetate, of polyacrylonitrile and polyvinyl alcohol, and of a copolymer of acrylonitrile with vinyl acetate and polyacrylonitrile. Solubility in acetone and spinning properties of the copolymers are examined. W.R.M.

### Heterogeneous Structure of Polyvinyl Alcohol Filament

H. Furuya

*J. Soc. Textile Cellulose Ind. Japan*,**12**, 193-197 (March 1956)

In an earlier paper (*ibid.*, **11**, 316 (1955)) the relationship between the heterogeneous structure of cross-section of polyvinyl alcohol filaments and the spinning conditions was investigated by the use of optical and electron microscopes. A model sample, in which skin and core regions could be observed separately, was prepared, in order to observe the detailed properties of both parts. It was concluded that the main factor in skin formation was the pressure of water, which diffused by osmosis during coagulation, since both optical and swelling anisotropies were observed in the gel formed by coagulation under no external force. The difference of adsorption abilities between the skin and the core was also estimated by measuring the rate of diffusion of iodine and dye through both parts. AUTHOR

### Improvement of the Tensile Recovery of Vinyon. VI—Mixed Acetalisation with Nonylaldehyde and Acetalyl Monosulphide, and Nonyl- and Benzaldehyde

N. Mori, K. Sakurai, T. Tanaka, and N. Nakamura

*J. Soc. Textile Cellulose Ind. Japan*,**12**, 245-248 (April 1956)

Vinyon fibres having good elastic recovery and better resistance to heat under dry and wet conditions can be obtained by acetalisation with acetalyl monosulphide before or after nonalaldehyde. In the mixed acetalisation with nonylaldehyde and acetalyl monosulphide, and also nonyl- and benzaldehyde, the effects of such acetalisation on the properties of the fibres depend upon the reaction conditions. AUTHORS

### Acetalisation of Polyvinyl Alcohol Fibre with Higher Aldehydes. I—Acetalisation of Heat-treated Polyvinyl Alcohol Fibre with Several Higher Aldehydes

K. Tanabe and K. Ohno

*J. Soc. Textile Cellulose Ind. Japan*,**12**, 258-264 (April 1956)

In order to improve the resilience of vinyon by introducing bulky side-chains, heat-treated polyvinyl alcohol

fibres are acetalised with several higher aldehydes, viz. butyr-, isovaleric, heptyl, lauryl, benz-, and  $\alpha$ -naphthoic aldehyde, and their properties, particularly tensile recovery, are examined. Their dry and wet heat resistances are somewhat inferior, their dry tenacities and elongations are approximately equal, and their wet tenacities are rather superior, to those of formalised fibres respectively, with the exception of fibres acetalised to higher degrees of acetalisation with higher aldehydes. Their Young's moduli are greater and their initial elastic loads and elongations are higher than those of formalised fibres, and these tendencies are particularly marked with benzalised fibres. The higher the aldehyde used and the greater the degree of acetalisation, the higher the tensile recovery in the range of relatively low extension, and inversely the lower in the range of higher extension. By benzalisation the best recovery and tensile recovery-extension curve similar to cellulose acetate are obtained. These relationships between tensile recovery and extension seem to be explicable in terms of steric hindrance due to side-chains introduced in amorphous portions of the fibre. The dyeability of fibres acetalised with these higher aldehydes is very poor compared with that of formalised fibre.

AUTHORS

### Polyvinyl Chloride Fibre

H. Sobue, K. Washimi, and M. Takahashi

#### IV—Polyvinyl Chloride Fibre containing Dinitronaphthalene as Plasticiser

*J. Soc. Textile Cellulose Ind. Japan*,**12**, 182-185 (March 1956)

Polyvinyl chloride (P.V.C.) fibres containing dinitronaphthalene (D.N.N.) are manufactured by dry spinning a soln. of P.V.C. in acetone and  $\text{CS}_2$ , and the fibres are drawn up to 400% elongation at 100°C. The stress-strain curves of P.V.C.-D.N.N. fibres thus obtained are measured by means of an Instron tensile tester. From these results, it is found that the strength and the elastic properties of P.V.C. fibres containing 5-10% D.N.N. are greater than those of pure P.V.C. fibres, but the elongation of the former is smaller. These properties are maintained over a wide range of temperature, but at about 100°C. the deformation and the shrinkage of pure P.V.C. fibres are smaller than those of D.N.N.-containing fibres, whose permanent deformations are, however, comparable with those of the pure P.V.C. fibres.

#### V—Solubility of Polyvinyl Chloride

*Ibid.*, 185-187

The swelling of P.V.C. films having thermal "bridging" is measured in acetone, methyl ethyl ketone, methyl isobutyl ketone, acetophenone, cyclohexanone, tetrahydrofuran, and mixtures of these ketones and  $\text{CS}_2$ . From these results, the parameters of the polymer-solvent interaction  $\mu$  are calculated. The viscosities of dilute solutions of P.V.C. in methyl isobutyl ketone, acetophenone, and cyclohexanone are measured, and the values of  $[\eta]$  are calculated. AUTHORS

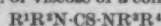
PATENTS

### Thick-skinned Regenerated Cellulose Filaments

Courtaulds

BP 748,147

Filaments having a thicker skin than usual are obtained by extrusion of viscose to which has been added < 0.1 millimole per 100 g. of viscose of a compound of formula—



( $\text{R}^1$  and  $\text{R}^2 = \text{H}$ , aliphatic or Ar;  $\text{R}^3$  and  $\text{R}^4 = \text{Ar}$ ), e.g. thiocarbonyl, in an aqueous bath containing  $\text{H}_2\text{SO}_4$ ,  $\text{Na}_2\text{SO}_4$ , and  $\text{ZnSO}_4$ .

### Water-soluble Polyvinyl Alcohol Yarn

DuP

USP 2,716,049

A solution of an 82-94% saponified polyvinyl ester is extruded into a conc. aq. sulphate bath, and the resulting yarn washed free of sulphate with an aq. soln. of a volatile salt in presence of boric acid and finally dried at a temperature which removes the volatile salt as well as the water. The resulting yarn, while readily soluble in water, withstands textile processing under normal humidity conditions. C.O.C.

### Readily Dyed Acrylonitrile Polymers

FH

BP 748,393

Acrylonitrile polymers containing acid imide groups, on being treated with polyvalent amines containing at least one amino group capable of being acylated, are given good affinity for acid and mordant dyes. C.O.C.



### Improving the Dyeing Properties of Polyester Fibres

ICI BP 748,944  
Treating Terylene or the like with an organic compound containing at least one secondary or tertiary amino group and one or more primary amino groups and/or one or more alcoholic hydroxy groups at 50–220°C. imparts increased affinity for dyes. Thus 140-denier Terylene yarn is passed at 40 yd./min. through 50% aq. triethanolamine at 25°C. and dried by passing for 5–10 sec. round a drum at 100°C. It is then baked at 165°C. for 20 min., rinsed for 5 min. each in ethyl alcohol and water, and dried. The treated yarn (10) dyed for 1 hr. in an aqueous suspension (500) of 4-nitro-4'-N-hydroxyethyl-N-ethylaminobenzene at 95°C., and then soaped, was dyed a scarlet which was 30 times as deep as that produced with an untreated yarn similarly dyed. C.O.C.

### Improving the Mordanting and Dyeing Properties of Polyester Fibres

Distillers Co. BP 749,456  
Treating polyester fibres with the vapours of sulphuric acid, sulphur trioxide or chlorosulphonic acid much increases their affinity for metallic mordants. C.O.C.

Kinetic Investigation of the Reaction between Carbon Disulphide and Caustic Soda Solution (III p. 385)

Colloid Chemical Studies on Starching Materials. I—Structural Viscosity of Polyvinyl Alcohol Solutions (X p. 402)

Fibre Science and Leather Science (XII p. 405)

Macromolecular Compounds (XIII p. 406)

Surface Structure of Wool and its Components revealed by Metal Shadowing (XIV p. 410)

## VII—DESIZING; SCOURING; CARBONISING; BLEACHING

### Critical Micelle Concentrations of Polyoxyethylated Non-ionic Detergents

L. Hsiao, H. N. Dunning, and P. B. Lorenz  
J. Phys. Chem., 60, 657–660 (May 1956)  
Critical micelle concentrations (C.M.C.) increase with ethylene oxide chain length and decrease with increasing concn. of electrolyte. The effect of chain length on C.M.C. is expressed by—

$$\ln (\text{C.M.C.}) = 0.056R + k$$

( $R$  = average number of ethylene oxide units;  $k$  is a constant depending on the electrolyte). These detergents form hydrophilic micelles with associated weak positive charges. W.R.M.

### Effect of Temperature on Micelle Formation and Solubilisation in Benzene

A. Kitahara  
Bull. Chem. Soc. Japan, 29, 15–19 (Jan. 1956)

### Open-width Bleaching of Cotton Piece Goods

P. J. Wood  
Amer. Dyestuff Rep., 45, 277–281 (7 May 1956)  
Desizing and bleaching in continuous open width are successfully carried out on the laboratory scale and a machine is proposed on the following lines—a desizing tank with open steam heating and final roller expression to give an uptake of 100% liquor, a horizontal slit-type steamer, and hot water in an open-width scoper. Three NaOH-impregnation tanks follow, the final squeeze roller (set to give 80–90% liquor in the fabric) being inside the next steamer to give a water seal, thus preventing ingress of air and consequent oxycellulose formation. A similar seal safeguards the far end, where the fabric passes through a hot water wash, a  $\text{CO}_2$  wash, and another hot water wash. It is safer to neutralise with  $\text{CO}_2$  than with  $\text{H}_2\text{SO}_4$ , and the by-product should be valuable. The peroxide bleacher is of similar construction with a sealed steamer. J.W.B.

### Action of Alkaline Hypochlorite on Methyl 2-O-Methyl- $\alpha$ -D-glucopyranoside

R. L. Whistler and S. J. Kazeniac  
J. Org. Chem., 21, 468–469 (April 1956)  
The intermediates formed produce, on acid hydrolysis, up to 0.54 mole of glyoxal, suggesting preferential oxidation at the  $\text{C}_2$  and  $\text{C}_4$  positions. H.E.N.

### Rag Bleaching, with especial reference to the Use of Hydrogen Peroxide

F. J. T. Harris  
Proc. Tech. Sectn. British Paper & Board Makers' Assoc., 36, 639–653 (Dec. 1955)

Attempts to increase the throughput of rag boilers and washers and, if possible, to improve the brightness of the bleached rags did not result in any major improvements when the established methods of hypochlorite bleaching, sodium chlorite, or hydrogen peroxide at about 3% consistency were applied. Good results were obtained, however, by a combination of bleaching and scouring, i.e. by cooking the rags under alkaline conditions with a 1–1.5 vol.  $\text{H}_2\text{O}_2$  bleach bath for about 2½ hr. at pressures up to 40 lb./sq.in. K.W.

Behaviour of Fabrics finished with Nitrogen-containing Resins towards Chlorine-containing Bleaches (X p. 402)

## VIII—DYEING

### Dyeing in a Fluidised Bed of Sand or Glass Beads

V. B. Chipalkatti  
J. Sci. Ind. Research (India), 14 B, 540–542 (1955); Chem. Abs., 50, 8798 (10 May 1956)

Direct, sulphur and vat dyes are padded into the goods, which are then passed for 5–20 sec. through the fluidised bed of sand or glass beads. Quick transfer of heat, evaporation of water and fixation of dye occur simultaneously, and effective temp. and dye concn. gradients are created across the fibre cross-section which help to increase rates of diffusion and fixation of dye. C.O.C.

### Ultrasonic Tanning and Dyeing

F. Gutman  
J. Brit. Inst. Radio Eng., 15, 357 (1955); J. Soc. Leather Trades Chem., 40, 113 (March 1956)  
A review of work in this field, giving 37 references. C.J.W.H.

### Improvement of the Rubbing Fastness of Azoic-dyed Fabrics

AATCC Piedmont Section  
Amer. Dyestuff Rep., 45, P 286–P 308 (7 May 1956)  
Studies are made of the effects of variables in azoic dyeing, including—coupling pH, coupling temperature, rinsing before coupling, steaming before coupling, soaping methods, addition of alcohol to the coupling bath, rubbing behaviour of various dyeing combinations, and the effect of subsequent finishes. No simple remedy is found, but several contributory causes are defined. Coupling pH has no pronounced effect between pH 3 and 8, though the shade itself may be affected outside the range 3.5–5.0. A pH of 4.5 is best, and heavier and more uniform dyeings are obtainable after intermediate drying. Coupling temp. has no effect over the range 32–120°C. *iso*-Propyl alcohol, recommended for stabilising coupling baths, has no noticeable effect on rubbing fastness. Steaming prior to coupling gives a slight improvement. The temperature of the rinsing bath before soaping does not affect rubbing fastness, but causes tarring if too high. Brushing to remove loosely held surface particles leads to improved fastness. Alkali-soap mixtures for scouring at higher temp. are better than synthetic detergents or plain water. Examination of a number of azoic combinations shows marked differences in rubbing fastness and indicates the need for careful selection. Only a few finishing agents were found which did not reduce fastness to dry rubbing: a silicone water-repellent had no influence, while the greatest reduction was caused by a finish comprising 2% of a dispersion of colloidal silica. Other water-repellents and fatty softeners adversely affected fastness to dry rubbing, but the effect of finishing agents on fastness to wet rubbing was much less pronounced. J.W.B.

### Physicochemical Studies of the Dyeing Process. I—Theory of Dyeing Equilibrium and Time of Half Dyeing of Direct Dyes on Viscose Rayon

M. Nakagaki  
Bull. Chem. Soc. Japan, 29, 201–208 (Jan. 1956)  
A general treatment of the theory of dyeing equilibrium. An equation expressing equilibrium from a finite bath is obtained, to which data by other workers can be fitted. It is found that rates of dyeing may be expressed by the simple relation—

$$\frac{x}{x_{\infty}} = \frac{1}{2} \sqrt{\frac{t}{T}}$$

( $x$  = dye adsorbed at time  $t$ ;  $T$  = time of half dyeing).

A.J.

### Package Dyeing. II—Effect of Liquor Ratio on Direct Dyeing

W. Armfield, J. Boulton, and J. Crank

J.S.D.C., 72, 278–286 (June 1956)

Package dyeing has been carried out on a laboratory apparatus with a number of dyes, each in 6 and 20 volumes, with enough sodium chloride added at the commencement of dyeing to give roughly the same final exhaustion at the two liquor ratios for any particular dye. After 30 min., with the same rate of flow, the 6-vol. dyeing was always more level than the corresponding 20-vol. dyeing. Special consideration is given to a class C dye which without added salt has a very high exhaustion in low volumes, due to the salt contained in the dye. Here also the 6-vol. dyeing was more level than the 20-vol. dyeing after 30 min., although the former package was more than twice as heavily dyed. These observations have been confirmed by dyeing commercial viscose rayon cakes on a laboratory cake-dyeing machine with single dyes and mixtures. The advantage gained by working in low volumes is shown to be due to an increase in the dyebath cycle rate, and an expression is given showing the relationship of this to yarn weight and rate of flow. The variation in yarn weight in bulk cake-dyeing is considered in so far as it affects liquor ratio and dyebath cycle rate. In the second part of the paper the results are examined in the light of a theoretical model of package dyeing previously described (*ibid.*, 68, 109 (1952)). It is found that, when certain parameters are chosen to give a fit with measured skein-dyeing curves, the main conclusions about package dyeing based on the experimental results are substantiated by the model. At the same time, more information is provided on the rate of exchange of dyes between liquor and yarn within the package, and on the influence of the three basic parameters on which package-dyeing behaviour depends.

AUTHORS

### Electron-microscopical and -diffraction Study of Solutions of Benzopurpurin 4B

S. Shishido

J. Fac. Sci., Niigata Univ., Ser. I, 2, (1), 1–15 (1955);

Chem. Abs., 50, 5366 (25 April 1956)

Electron-microscopical and -diffraction study of solutions of Benzopurpurin 4B containing excess of NaCl shows that presence of dye micelles retards the growth of certain crystal planes of NaCl and that the NaCl acts as a combining agent that facilitates the growth of dye micelles.

C.O.C.

### Interaction between Chlorazol Sky Blue FF and Chrysophenine G in Aqueous Solution

A. N. Derbyshire and R. H. Peters

J.S.D.C., 72, 268–277 (June 1956)

The absorption spectra of mixtures of Chlorazol Sky Blue FF and Chrysophenine G in aqueous solution have been measured at room temperature and at 60°C. Spectra of regenerated cellulose sheet dyed with the two dyes from mixture dyebaths have also been determined. Paper and column chromatography on mixtures of the two dyes confirm the formation of complexes, deduced from the spectral measurements. The heat of formation of the 1:1 complex of the two dyes, measured by direct calorimetry, is –15 kcal./mole. Finally, equilibrium adsorption of the two dyes by cotton from mixture dyebaths has been determined. It is concluded that formation of a 1:1 complex occurs in aqueous solution to an appreciable extent even at 90°C. This complex formation explains in a semi-quantitative way the change in dye adsorption compared with that of the same dyes when applied singly. It is very probable that failure of dyeing data on single dyes to predict the behaviour of the same dyes in mixtures is due to interaction of this kind between the dyes in solution.

AUTHORS

### Control of Sodium Hydrosulphite in Vat Dyebaths

W. J. Marshall

J.S.D.C., 72, 201–210 (May 1956)

In vat dyebaths, the basic information required for control of the hydrosulphite is the rate at which it must be replaced and the properties of the dye which determine the

minimum concentration of hydrosulphite necessary for successful dyeing. For this reason the rate of oxidation of hydrosulphite solutions has been measured over a wide range of conditions, and the results have been related to typical practical systems. The reduction properties of vat dyes are discussed with reference to their reduction potentials, and it is shown that electrometric methods can be satisfactorily applied to dyebath control only in special circumstances. The overriding difficulty is the insensitivity of the electrode to changes in hydrosulphite concentration and the variable speed—always low—with which a steady potential is reached. It is concluded that the most satisfactory method of control depends on an initial quantitative study and thorough understanding of the hydrosulphite requirements of the dyeing system. Methods of measuring hydrosulphite concentration suitable for dyehouse use are briefly described.

AUTHOR

### Eastofix Dyes

J. M. Straley and D. G. Carmichael

Amer. Dyestuff Rep., 45, P 260–P 262 (23 April 1956)

Recipes are briefly given for dyeing, including jig-dyeing, and printing cellulose acetate with Eastofix (TM) metalisable dyes.

J.W.B.

### Wool Dyeing—Loss in Weight and its Reduction

J. Bauer

Amer. Dyestuff Rep., 45, P 253–P 259 (23 April 1956)

In blends with new fibres wool is being subjected to more severe chemical treatments than ever before, wool-Dacron union dyeing being a case in point because of the longer dyeing cycle—in some instances up to 14 hr. Loss of protein and consequent reduction in wearing properties can be and often are very serious. Figures are presented of weight loss under various conditions, e.g. different times of boiling in different solutions, and it is suggested that weak acids are a safeguard, especially if the pH is maintained near the isoelectric region. The use of formaldehyde (about 0.5–3.0% on wt. of fibre) is of value with sulphuric acid dyeings; and high-temperature dyeings in the isoelectric region in presence of  $\text{CH}_2\text{O}$  offer a promising method of applying metal-complex acid dyes.

J.W.B.

### Colloidal Chemistry of Haematein Salts

N. I. Gimesi

Acta Biol. Acad. Sci. Hung., 6, 7–17 (1955);

Chem. Abs., 50, 6144 (10 May 1956)

Cellulose or silk fibres, which have an oriented microcrystalline structure, show great adsorption of haematein dye salts (I), probably because of the large surface offered. Molecular I penetrates and dyes the cell nucleus better than colloidal I. The neutral salts do not form dyes. Alkali hydroxides give molecularly dispersed, neutral I. Cu and Ba I have properties between those of molecularly and colloiddally dispersed dye. The salts are less soluble.  $\text{Al}_2(\text{SO}_4)_3$ ,  $\text{FeCl}_3$  and  $\text{K}_2\text{CrO}_4$  which hydrolyse completely, give molecularly dispersed I in acid solution. In neutral or alkaline solution, or upon electrophoresis, the I coagulates. The positive charge is obtained from the free phenol-like OH groups and the dissociated H from the sulphonated derivative.

C.O.C.

### Practical Aspects of Dyeing in the Burlington Beam Machine

P. L. Meunier

Amer. Dyestuff Rep., 45, P 327–P 330, P 336 (21 May 1956)

Methods, advantages, and disadvantages of dyeing fabrics of nylon, Orlon, Dacron, and cellulose triacetate on both open and pressurised beam-dyeing machines are discussed.

J.W.B.

### Dyeing of Orlon and Orlon Mixtures

I. M. S. Walls

J.S.D.C., 72, 261–268 (June 1956)

The physical characteristics of the acrylic fibre Orlon are described briefly. Disperse dyes, basic dyes, acid dyes applied by the cuprous ion technique, and indigoid and thioindigoid vat dyes can be used to dye this fibre, and the dyeing and fastness properties of each class of dye are described fully. The selection of dyes for specific purposes is discussed, and some advice on practical dyeing and finishing procedure included. The dyeing of mixtures of Orlon with wool, nylon, and cellulosic fibres is described, and the paper concludes with some comments on the dyeing properties of analogous European acrylic fibres.

AUTHOR

**High-temperature Dyeing of Dynel**

W. W. Rankin and T. H. Newman

*Amer. Dyestuff Rep.*, 45, 213-214 (9 April 1956)

The optimum temperature for dyeing dynel under pressure is 220°r.; negligible shrinkage occurs, though more takes place with some dyes than with others. High temperatures are particularly advantageous with expensive dyes. Levelness and fastness are as good as those obtained by dyeing at 210°r. Precautions must be taken to avoid channelling, a flow of 1½ gal.(U.S.A.)/min. per lb. of fibre being best. A few recipes are briefly given.

J.W.B.

**Substantivity of Azolic Dyes for Vinylon Fibres**

K. Yasuda and K. Konishi

*Repts. Ind. Research Inst., Osaka Prefect.*

7, (2), 62-66 (1955):

*Chem. Abs.*, 50, 6053 (25 April 1956)

Increase in temperature causes much greater increase in substantivity for Vinylon than it does in that for cotton. Maximum substantivity for Vinylon occurs at 40-60°C., but the depth of the dyeings increases with increase of temperature. This contradictory result may be related to the special structure of Vinylon and the mechanism of dyeing. An iodine titration method is described which measures substantivity much more accurately than does the precipitation method.

C.O.C.

**Dyeing of Blotting Papers**

B. Cornely

*Allgem. Papier-Rundschau*, 1020-1025 (1955):*Chem. Abs.*, 50, 6046 (25 April 1956)

An account of the dyeing of high-grade blotting papers made from cotton and/or purified bleached sulphite pulps. A list of suitable dyes, mostly direct dyes, is given together with a list of dyes whose use is to be avoided.

C.O.C.

**Colouring Butter with Synthetic  $\beta$ -Carotene**

H. Aust

*Milchwiss. Ber.*, 5, 85-92 (1955):*Chem. Abs.*, 50, 6098 (10 May 1956)

Butter coloured with synthetic  $\beta$ -carotene had no loss in flavour after storage at -2, 5, or 10°C. The faint red or green coloration often seen in butter coloured with bixin was not seen in samples coloured with the synthetic  $\beta$ -carotene.

C.O.C.

## PATENTS

**Overdyeing Stockings to produce Two-colour and Ombre Effects**

S. Bailey

USP 2,716,585

Ombre-dyed nylon stockings having welt and afterwelt regions of contrasting colour to the rest of the stocking, the transition from one colour to the other being imperceptible, are obtained by first dyeing the stockings in the normal manner. They are then dried, preferably on forms, assembled in lots of multiple pairs, e.g. one dozen pairs, and suspended by their tops from between clamp-type hangers. The hangers are then hung from a rack, and the stockings lowered into a second dyebath at a controlled speed until the level of the dye liquor is about 4 in. below the afterwelts of the stockings. They are then lifted out of the liquor at the same speed. This is repeated 2-5 times over a period of 15 min. while the liquor is at 150-170°r.

C.O.C.

**Colouring Glass Fabrics**

R. W. Stenzel, H. Plaut, and G. G. Buchanan

USP 2,716,618

The cloth is heated at least twice in presence of inorganic materials which combine to precipitate a coloured compound on the cloth. Thus the cloth is treated with an aq. soln. of a metallic salt and then heated to 900-1200°r. to form a layer of the metal oxide on it. It is then cooled, treated with a second solution of a material which yields a coloured compound with the oxide, and again heated to 900-1200°r.

C.O.C.

Effect on Dye Structure on the Dyeing of Orlon 42 with Azo Dyes (IV p. 386)

Improving the Dyeing Properties of Polyester Fibres (VI p. 399)

Microscopical Studies on Wool Dyeing—Preparation and Use of Longitudinal Sections (XIV p. 411)

**IX—PRINTING**

## PATENTS

**Photographically Sensitive Sheet for Preparing Stencils**

C. Eckardt

BP 748,767

A method of preparing stencils particularly suitable for stencils for diffusion printing.

C.O.C.

**Fixing Dyes on Fabrics and Foils containing Cellulose**

Ciba

BP 748,699

The material is padded or printed with a dye containing a readily hydrolysed acyl group, and is then treated with an aqueous alkaline medium in presence of hydrogen peroxide. Thus cotton fabric is printed with a paste made of 78 parts of the reaction product of 2 mol. of *m*-sulphobenzoyl chloride with 1 mol. of the azo dye 2:5-dichloroaniline  $\rightarrow$  1-(2-hydroxy-3-naphthoylamino)-2-methoxybenzene, water (322), thiodiglycol (100) and neutral starch-tragacanth thickening (500). The printed fabric is dried, steamed for 5-10 min. in a Mather & Platt ager, heated at room temperature for 1-2 min. in a bath of water (1000 parts by volume), Na<sub>2</sub>CO<sub>3</sub> (50), NaOH (4) and Na perborate (4). It is then rinsed with cold water, treated with an enzyme and soaped at the boil for 10-15 min. This gives a strong bright scarlet print.

C.O.C.

**Preparing Polyethylene for Printing**Dup<sup>r</sup>

USP 2,715,075-6

The adhesion of printing inks to polyethylene is improved if the polyethylene before being printed is treated at 150-325°C. with ozone in presence of a halogen, a hydrogen halide, or nitrous oxide, and, preferably, in presence of ultraviolet radiation.

USP 2,715,077

The polyethylene is treated with nitrous oxide at 150-325°C. preferably in presence of ultraviolet radiation.

C.O.C.

**Making Differently Coloured Marks with a Single Fluid**

National Cash Register Co.

USP 2,714,074

A fluid containing an ink and a colour-forming component is applied to material which may or may not be sensitised either wholly or in part to react with the colour-forming component. Thus an ink containing Sudan III and Crystal Violet lactone yields a red mark on unsensitised paper and a purple mark on paper sensitised with attapulgite.

C.O.C.

**Contact Printing Emulsion for Application to Glass, Vinyl Plastics, or the like**

Direct Reproduction Co.

USP 2,716,060-1

**Photographic Dye Transfer Process**

Eastman Kodak Co.

USP 2,713,305

A silver halide image is treated with a basic dye in presence of a water-soluble halide salt, e.g. NaCl. The dyed image is then used to make a number of imbibition dye prints by pressing it against a mordanted receiving sheet in presence of a soluble silver salt. The function of the silver salt is to reverse the charge on the silver halide and allow the dye to diffuse to the receiving sheet.

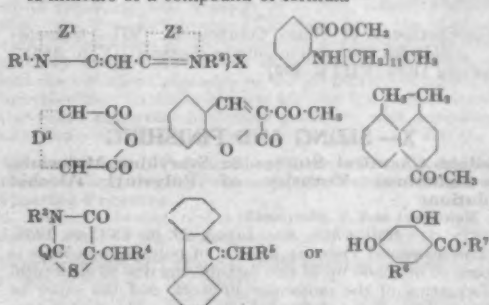
C.O.C.

**Ultraviolet-radiation-absorbing Compounds and their Use in Colour Photography**

Kodak

BP 748,189

A mixture of a compound of formula—



(R<sup>1</sup> and R<sup>2</sup> = Alk; R<sup>3</sup> = H, Alk or Ar; R<sup>4</sup>, R<sup>5</sup> and R<sup>7</sup> = Ar; R<sup>6</sup> = Alk, O-Alk, or if R<sup>7</sup> contains a solubilising group







### Impregnating Cellulose Materials with Thermosetting Resin to Impart Dimensional Stability

Monasanto USP 2,715,078

The material, preferably of regenerated cellulose, is treated in a liquor at 100–200°r. containing 0.2–2.0% (on the wt. of material) of a cationic surface-active agent until the liquor is exhausted. The material, which if desired may be rinsed and/or dried, is then treated in a long liquor at 115–180°r. containing an unpolymerised alkylated melamine-formaldehyde, urea-formaldehyde or alkylated urea-formaldehyde condensate until the liquor is exhausted. The liquor may contain an alkali-metal salt or alkaline-earth-metal salt to assist in exhausting the condensate from the liquor, but it must be free of acid or latent curing catalysts. The material is then dried, and finally baked at 250–350°r. This gives good resistance to shrinkage on washing without appreciable lowering of tensile strength. C.O.C.

### Imparting to Cellulosic Textiles Water-repellency and Ability to Recover from Creasing

ICI BP 748,182

The materials are treated in either order or simultaneously with a water-repellent agent of the type described in BP 583,031, preferably stearamidomethoxycetic acid, and an agent which imparts ability to recover from creasing, e.g. a urea- or melamine-formaldehyde precondensate. When used in the same bath the mixed solutions remain stable and no decomposition products are left on the materials after they are baked. C.O.C.

### Rendering Cellulosic Materials Flame-resistant

DuP BP 748,165

The material is impregnated with an aqueous monobasic acid solution of titanium and tervalent antimony diluted with an initially stable, water-soluble, volatile, organic solvent. The weight ratio of Ti to Sb is  $\geq 10$ , the atom ratio of Sb to Ti is  $\geq 2$ , and the amount of organic solvent is preferably  $\geq 70\%$  by wt. Thus 80 parts by wt. of an aqueous solution containing  $\text{TiO}_2$  (160 g.),  $\text{Sb}_2\text{O}_3$  (292 g.),  $\text{ZnO}$  (32 g.) and  $\text{HCl}$  (11 mol.) per litre was diluted with water (10 parts) and isopropyl alcohol (10). A strip of 6-oz. cotton cloth was impregnated in this solution, given a moderate squeeze, exposed to moving air for 5 min., stood for 15 min., immersed in 15% aq.  $\text{Na}_2\text{CO}_3$ , washed and dried. After repeated washings the treated cloth showed negligible after-flame and afterglow characteristics. C.O.C.

### Coated or Laminated Sheets

Chemische Werke Albert BP 747,969

Sheet material, e.g. cloth or paper, is either impregnated with a thermosetting moulding resin or evenly coated with a thin layer of powdered or fibrous thermosetting moulding resin. It is then treated with the vapour of a solvent until the resin is swollen without any being reduced to liquid. The whole is then hot-pressed under low pressure to bond the applied resin to the material and the plies, if any, together. C.O.C.

### Shrinking Knitted Fabric

S. Anderson BP 747,981

The fabric is fed between two elastic webs formed by helical springs. These webs are driven in the same direction and run parallel to one another. During their travel the springs contract so as to compress the fabric longitudinally while at the same time it is subjected to steam and heat. C.O.C.

### Imparting Dimensional Stability to Tubular Knitted Fabric

F. R. Redman USP 2,714,756

The fabric is expanded widthwise, while sufficiently free lengthwise to relieve the distorted stitch loops. It is then relaxed in all dimensions and finally steamed while completely relaxed. C.O.C.

### Bonding Yarns to Polyvinyl Chloride Compositions

Courtaulds BP 749,701

Much better adhesion is obtained between regenerated cellulose yarns and polyvinyl chloride compositions if the yarns are treated with *m*-phenylenediamine, which may be allowed to oxidise before the yarns are brought into contact with the polyvinyl chloride compositions. C.O.C.

### Bonding a Polyethylene Film to a Fibrous Web

Dobackman Co. USP 2,714,571

### Reducing the Felting Power of Wool

Albany Felt Co. USP 2,714,051

Wool is treated for  $\geq 1$  sec. with nascent bromine. Thus wool is treated with an alkali-metal bromate (1 mol.) and an alkali-metal bromide (5 mol.) and then with an acid so as to convert very gradually the bromate and bromide ions absorbed by the wool into bromic and hydrobromic acids. These acids react with each other rapidly to produce nascent bromine, which, in turn, reacts with the clean wool within 1 sec. Careful control of (1) the rate of production of the bromine and (2) the rate of its reaction with the wool prevent evolution of free bromine or hypobromous acid harmful to the wool. C.O.C.

### Impregnating Fibrous Material with a Viscous Liquid

V. E. Yarnley, A. G. Goodchild, and A. M. Hutcheson BP 748,339

When impregnating fibrous material with a viscous liquid, e.g. glass cloth with a resin syrup, the impregnated material is vibrated so as to break down the surface tension of the resin syrup and part the fibres, so that the syrup may flow into the voids between the fibres. This prevents any voids being left in the finished material. C.O.C.

### Recovery from Bending of Wires and Filaments of Polyethylene Glycol Terephthalate (VI p. 398)

Improvement of the Rubbing Fastness of Azoic-dyed Fabrics (VIII p. 399)

## XI—PAPER AND OTHER CELLULOSIC PRODUCTS

### Corrosion of Aluminium in Contact with Paper

T. H. Angel  
Proc. Tech. Sectn. British Paper & Board Makers' Assocn., 36, 499–508 (Dec. 1955)

Moisture and electrolytes cause pitting or filiform corrosion of annealed Al foil, which is more severe at the sides of the sheet than at the centre, chlorides being the most corrosive compounds normally found in paper. The surface of freshly annealed Al becomes hydrophobic in contact with paper. Paper having a moisture content of 7% will be safe if the content of chlorides (as  $\text{NaCl}$ ) is  $\geq 0.1\%$ , water-soluble sulphate (as  $\text{Na}_2\text{SO}_4$ )  $\geq 0.2\%$ , and the pH  $\sim 5$ . If higher moisture contents are expected, chlorides and sulphates must be lower. Inhibitors have little effect if the chlorine content is high. K.W.

### Ink-Paper Relationships in Printing

R. R. Coupe  
Proc. Tech. Sectn. British Paper & Board Makers' Assocn., 36, 669–682 (Dec. 1955)

Recent work carried out on ink transfer and penetration, and on the influence of paper on autoxidation drying, is discussed. K.W.

### Varnishing Characteristics of Coating Clays and Pigments

J. C. Rice

Tappi, 39, 43–46 (1956);  
Chem. Abs., 50, 6087 (25 April 1956)

Comparison of the optical properties of overprint varnish films applied to coatings made from 8 typical clays and white pigments over a range of 5–25% adhesive shows that—(a) fine-particle-size U.S. clays have the highest varnish resistance of the materials tested, (b) a unique contrast exists between the behaviour of the resistant and the absorbent pigments as the proportion of adhesive increases, and (c) the brightness and the ink receptivity of the white pigments drop more gradually with increased adhesive content than do those of the clays. C.O.C.

### Mercerisation of Pulp in Sheet Form. III—Investigations on the Steeping Process by the Determination of Floating Pressure

A. Iuchi, T. Owashi, and A. Miyamoto  
J. Soc. Textile Cellulose Ind. Japan, 12, 89–95 (Feb. 1956)

### Fractionation of Cellulose with Sulphuric Acid

C. Simionescu and E. Calistru  
Faserforsch. und Textiltech., 7, 171–175 (April 1956)

Cellulose is pretreated with 50% sulphuric acid and dissolved in 60–65% acid at  $-15^\circ\text{C}$ . Precipitation at

—15°C. is effected by dilution of the acid. The low temperature is said to reduce hydrolysis. W.R.M.

#### Cellulose Studies. XXI—Rate of Degradation of Cellulosic Materials to the Oligosaccharide Stage in Phosphoric Acid Solution

R. A. Martin and E. Pacsu

*Text. Research J.*, **26**, 192–204 (March 1956)

The rate of decrease in the number-average D.P. of viscose rayon (initial D.P. 384), regenerated hydrocellulose (initial D.P. 52), and native hydrocellulose (initial D.P. 288) in 85%  $H_3PO_4$  (1% soln., 21°C.) during a major part of the hydrolytic process has been determined. Samples taken at intervals were poured on to ice, when all materials having D.P. > 16 pptd. (primary ppt.). Centrifugates from primary ppt. were neutralized by passage through a column of Amberlite IR-4B and concentrated, when secondary ppt. (D.P. 7–16) were obtained. Aliquots of centrifugates from secondary ppt. were titrated with alkaline hypoiodite to determine total reducing power of water-soluble sugars present (A). Further aliquots were hydrolyzed to convert all oligosaccharides to D-glucose and titrated with hypoiodite (B). The ratio B/A gives the number-average D.P. of the saccharides present in the centrifugates. Number-average D.P. of primary and secondary ppt. were also determined by estimation of reducing end-groups. A combined number-average D.P. for each sample withdrawn at a particular time was calculated. From these D.P. values the rates of decrease of the combined number-average D.P. throughout the hydrolytic process were calculated. All three celluloses gave essentially identical results. The initially high values of the rate constant rapidly decreased to a minimum after the first 2–3 weeks of the reaction, and then gradually increased, finally approaching the value of  $k$  for cellobiose. The min. value of  $k$  found for hydrocellulose ( $0.7 \times 10^{-4}$  min.<sup>-1</sup>) is about 9% of the reported value for cotton cellulose and about 6% of the rate constant for cellobiose. It is suggested that the explanation of these results lies in the micellar behaviour of cellulose in solution rather than in the assumption of the presence of abnormal, easily hydrolysable units in the cellulose chains. J.C.F.

#### Studies on Viscose. XIX—Surface Structure of Regenerated Cellulose Film

M. Fujii and Y. Ono

*J. Soc. Textile Cellulose Ind. Japan*, **12**, 83–88 (Feb. 1956)

The effects of the degree of ripening of viscose, and of the concn. of acid,  $(NH_4)_2SO_4$ ,  $ZnSO_4$ , etc. in the coagulating bath, on the surface structure of regenerated viscose films were observed using an electron microscope. The smoothness or the roughness of the surface of films is affected, not only by the concn. of acid and salts in the coagulating bath, but also by the stability of the xanthate. For example, when a coagulating bath of high  $ZnSO_4$  content is used, a stable zinc xanthate film is formed, and the surface of the regenerated film becomes very rough owing to delayed decomposition. AUTHORS

#### Cornification of Cellulose and Reactivation of the Cornified Cellulose

R. Hasebe, K. Matsumoto, and H. Maeda

*J. Soc. Textile Cellulose Ind. Japan*, **12**, 203–207 (March 1956)

Measurement of the swelling of samples in glacial acetic acid yielded the following results—(1) In the dehydrating stage of drying, cornification develops increasingly after the water content has fallen below 50% in the case of native cellulose, or below 80% in the case of cellulose previously treated with 10% NaOH. (2) Cornification is appreciably inhibited by drying after replacing water with an organic liquid, and the smaller the surface tension, affinity for the hydroxyl group, and polarity of the liquid, the greater the inhibiting action of the liquid. (3) If cellulose not cornified during drying is placed in a room of moderate humidity, cornification takes place markedly during storage. (4) The cornified cellulose is reactivated by treating it with a suitable liquid; the greater the cohesive energy density of the liquid, the higher its activity as a reviving reagent. Acetic acid displays its reviving action only at moderately high temperature. (5) Mechanical grinding is very effective in the reactivation of the cornified cellulose. AUTHORS

#### Nature and Physicochemical Properties of Solutions of Cellulose in Cupreous Solvents

N. Gralén and J. Linderot

*Svensk Papperstidning*, **59**, 14–18 (15 Jan. 1956)

Light-absorption measurements have shown that the cellulose-Cu complex in cupriethylenediamine (I) solutions of cellulose is very probably of the composition  $[Cu en_2]^{2+}[Cu(C_6H_7O_2)]_n^{2-}$ , and that in cuprammonium (II) has a corresponding composition. The charge effects in sedimentation and diffusion are very small, as the cellulose-Cu complex is very little dissociated and the electrolytes present in ordinary I or II solutions depress the charge effects to 0.2–0.3%. Addition of  $NH_4SCN$  to reduce the charge effects is not suitable, as it causes the cellulose molecules to agglomerate. The partial sp. vol. of the cellulose-Cu anion is 0.532 in I and 0.508 in II; the mol. wt. of the complexes are 540,000 (I) and 500,000 (II); these results are much higher than those obtained by other methods, and the reasons for the discrepancies are discussed. K.W.

#### Thermal Degradation of Wood and Cellulose

A. J. Stamm

*Ind. Eng. Chem.*, **48**, 413–417 (March 1956)

#### Structural Changes of Alkali Cellulose during Ageing

K. Lauer and L. Skarik

*J. Polymer Sci.*, **20**, 397–404 (May 1956)

During ageing the sedimentation volume of the acidified fibres passes through a maximum, indicating a high degree of swelling. Xanthation shows that certain intermicellar bonds are broken, permitting better accessibility for NaOH and  $CS_2$ . The sedimentation maximum is always observed at D.P. of ~450–500, and viscose prepared with these alkali celluloses has a definite viscosity range. Determination of the sedimentation volume can be used as a method of finding the desired degree of ageing in filterability measurements. W.R.M.

#### Reaction between Cellulose and Heavy Water

J. Mann and H. J. Marrinan

##### I—Qualitative Study by Infrared Spectroscopy

*Trans. Faraday Soc.*, **52**, 481–487 (April 1956)

Liquid and vapour deuterations are studied, and it is shown that in addition to amorphous regions a limited proportion of crystalline regions can be deuterated. The reaction is much slower in the vapour phase, and all the amorphous regions can be deuterated without appreciable deuterium of crystalline regions. Crystallinity can therefore be estimated. Liquid-phase deuteration gives rise to a small amount of OD groups in crystalline regions which are very resistant to rehydrogenation. These resistant groups are formed by a change in the cellulose structure.

##### II—Measurement of Absolute Accessibility and Crystallinity

*Ibid.*, 487–492

A method is developed for determining the accessibility of cellulose to liquid heavy water. An attempt is made to avoid errors in previous work. A method is also developed for determination of absolute crystallinity in which the cellulose is deuterated with  $D_2O$  vapour until all amorphous regions are deuterated. The deuterium which has exchanged with the cellulose is then extracted with water, and the amount found by measuring the refractive index of the mixture. Advantages of this technique are discussed.

##### III—Quantitative Study by Infrared Spectroscopy

*Ibid.*, 492–497

A method is developed which enables the crystallinity of a cellulose to be estimated by infrared spectroscopy. Relative crystallinities agree reasonably well with those obtained by Hermans' X-ray method, but absolute crystallinities are about one-third lower. Measurements on Fortisan and cotton micelles prepared by acid hydrolysis show that these micelles contain appreciable amounts of amorphous OH groups. Estimates of the fraction of crystalline regions which can be deuterated with liquid  $D_2O$  have been made. W.R.M.

#### Chain-length Distribution of Cellulose Acetate.

##### I—Change in the Chain-length Distribution of Cellulose Acetates during Saponification

H. Sobue, K. Matsuzaki, and K. Yamamura

*J. Soc. Textile Cellulose Ind. Japan*, **12**, 100–104 (Feb. 1956)

By acetylation of cotton linters three cellulose acetates, of degree of acetylation 59.7, 57.4, and 54.9%, were

obtained. The first was fractionated with methylene chloride-ethanol, and the others with acetone-ethanol. The acetates were saponified with 20% ammonium hydroxide, and the nitrates made by nitration of the regenerated cellulose were fractionated with acetone-ligroin. In a comparison between the distribution curves of nitrates and of acetates, it was shown that the fractionation of the nitrates gave more precise results than that of the acetates themselves. In the distribution curves the high-D.P. portions decreased with the time of saponification. A non-uniformity coefficient increased in the early stage, but decreased as saponification proceeded. The experimental results were studied on the basis of the Beall-Jørgensen theory. It was assumed that hydrolysis of the glucoside linkage takes place at random in the initial stages of saponification, but not at random after the hydrolysis has proceeded.

AUTHORS

## PATENTS

**Cast-coated Paper**

J. D. Warren Co.

BP 747,107

Paper is first coated with an aq. mineral coating composition, and the coated surface is then fed continuously over the highly polished chromium-plated surface of a heated casting drum so as to maintain contact without relative movement between the two surfaces. A pool of water free of suspended solids is maintained in the nip between the two surfaces to prevent air from being entrapped in the nip, to wet the metal surface, and to ensure good contact between the surfaces. The water may contain small amounts of dissolved colloidal coagulating and/or solubilising agents, buffering agents, and release agents.

K.W.

**Coating Paper with Crystalline Saran**

Dow Chemical Co.

USP 2,716,074

A method of applying a crystalline saran in which the tendency of the coated paper to curl is overcome.

C.O.C.

**Regenerated Cellulose Sheet resembling Metal Foil**

DuP

USP 2,716,614

Incorporation into viscose of 20-65% by wt. of finely divided, heat-treated mica, 0-4% of stainless steel flake and 0-12% of colouring agent, regenerating the cellulose and impregnating the resulting sheet in an aq. bath containing 3-5-8-0% glycerol and 0-5% urea, the glycerol:urea :: 1-6:1, yields sheeting which looks like metal foil.

C.O.C.

**Properties of Solutions of Cellulose Xanthates of Low**

Degree of Substitution (VI p. 396)

Dyeing of Blotting Papers (VIII p. 401)

Coated or Laminated Sheets (X p. 403)

**XII—LEATHER; FURS; OTHER PROTEIN MATERIALS****Fibre Science and Leather Science**

H. Phillips

*Chim. et Ind.*, 75, 263-274 (Feb. 1956):*Rev. Tech. Ind. Cuir*, 48, 111-124 (June 1956)

The chemical constitution and the physical structure of collagen are reviewed: the fibrous character of collagen is emphasised, and analogies with common textile fibres are drawn. Evidence of alternating regions of crystallinity and amorphousness in collagen fibrils is reviewed; the crystalline regions are "auto-protected", whilst the amorphous regions are protected and reinforced by tannage. The degree of crystallinity of collagen may be determined either (1) from the sorption isotherm (Hailwood and Horrobin, *Trans. Faraday Soc.*, 42, 84 (1946)), or (2) by deuteration (method of Hill and the author) through prolonged exposure to D<sub>2</sub>O vapour, the labile hydrogen of peptide, amino, guanidyl, amide, and carboxyl groups being replaced by D. The latter method gives much lower values than the former; the difference is serious, but attention is drawn to the complexity, sensitivity, and the importance of the history of the fibrous system concerned. The action of tannage upon the amorphous regions of the fibres occurs in two stages—(1) association between the tannins and the exposed molecular chains of collagen; (2) association between the fixed tannins and further tannin molecules, which continues until all the accessible free space is occupied. At this stage tannage is complete,

and 100 parts of fibre retain ~50 parts of tannin. Measurements of the rate of tannage with 10 different tannins (Stather and Lauffmann, *Colloquium*, 75 G, 420 (1935)) have shown that tannage is virtually complete within 22 days. Roddy (*J. Amer. Leather Chem. Assoc.*, 38, 143 (1943)) has effected complete tannage within 48 hr. by means of a conc. soln. of quebracho in acetone followed by fixation of the tannin by steeping in water, or in a soln. containing H<sub>2</sub>SO<sub>4</sub> (1%) and NaCl (10%), in each case for 24 hr. The resulting degree of tannage (47-48) was increased (to 70-80) by subsequent tannage in the works. Pretannage liming causes slow separation of the molecular chains in the amorphous regions of the collagen, thus increasing the "water-soluble" tannin (that removable from the tanned leather by a prolonged extraction with water, corresponding to stage (2) above); the fixed tannin (that which resists water extraction, corresponding to stage (1) above) is virtually unaffected, showing that no opening up of the crystalline regions occurs. Tannage in weak acid soln. at an elevated temp. ("hot-pitting") swells the amorphous regions, and even opens up the crystalline regions to a small extent; this method gives hard and impermeable leathers suitable for soiling. Modification of the crystalline regions must be minimised to avoid weakening the leather. J.W.D.

**Helical Nature of the Collagen Fibril**

R. Reed, M. J. Wood, and M. K. Keech

*Nature*, 177, 697-699 (14 April 1956)

Evidence is discussed to support the view that the helical structures observed under the electron microscope are best accounted for in terms of an architecture of the collagen fibril based on a system of filaments in a regular cross-helical arrangement. Since the polypeptide chains in the fibril probably differ in chemical composition, these fibrils (i.e. bundles of polypeptide chains) may consist almost entirely of one type of chain, and they may thus also differ in their total chemical composition. These helical structures have been observed at electron microscope level, and it is still possible for the filaments to have an internal coiled-coil structure, as suggested by recent X-ray diffraction studies. There are nine electron micrographs, and a diagram of the proposed cross-helical structure.

J.W.D.

**Structure of Collagen**

G. N. Ramachandran

*Nature*, 177, 710-711 (14 April 1956)

The structure proposed by Ramachandran and Kartha (*Nature*, 176, 593 (1955)) was criticised by Rich and Crick (*ibid.*, 176, 915 (1955)); the author replies to these criticisms, and also comments on a proposed structure due to Cowan, McGavin, and North (*ibid.*, 176, 1062 (1955)). Attention is drawn to a further publication on the same subject, by Ramachandran and Kartha (*Proc. Indian Acad. Sci.*, 42, 215 (1955)), in which the possibility of two oppositely coiled structures is recognised; these, produced by rotating the minor helices about their own axes through a positive or negative angle, are termed *plus* and *minus* structures respectively. The *minus* structure is preferred (the reasons are given in detail), although both Rich and Crick, and Cowan *et al.*, have favoured the other possibility. Although there are many points in favour of the *minus* structure, a second set of hydrogen bonds cannot be formed, and a number of imino hydrogens are systematically not bonded; they may, however, be bonded to a carbonyl oxygen in another chain of the same triple chain through a water molecule.

J.W.D.

**Comparison of Methods of Degreasing employing Anionic and Non-ionic Accessory Agents**

R. F. Innes and K. G. A. Pankhurst

*J. Soc. Leather Trades Chem.*, 40, 99-110 (March 1956)

The use of a non-ionic surfactant of the long-chain phenol-ethylene oxide type (I), both as an accessory in the paraffin degreasing process and as an aq. emulsifier for removing grease from wet sheep pelts, has been studied, and the results have been compared with those obtained by using sulphated amyl oleate (II), and paraffin as recommended by the British Leather Manufacturers' Research Assocn. The results, which are based on laboratory tests and works-scale trials in three tanneries (all these are fully reported), indicate that solvent degreasing is superior to a purely aq. process. The efficiency of paraffin degreasing is enhanced by the use of I or II in addition, I being slightly the more effective. Neither I nor II in aq.



soln. (without paraffin) is satisfactory when the skins contain intact fat cells. J.W.D.

### Modern Resinous Tanning Materials, their Properties and Uses

R. Hayden and J. Plapper

*Leder*, 6, 215-219 (1955):

*Chem. Abs.*, 50, 6825 (10 May 1956)

A review of the preparation of melamine-dicyandiamide condensates. Whereas the classical urea and melamine resins are fixed in leather mainly by increase in molecular size, the newer melamine-dicyandiamide resins are absorbed by leather as cationic or anionic substances in the same way as other high-molecular ionic materials, e.g. dyes. They can also be deposited by precipitation with salts, acids or oppositely charged substances. Their use in tanning is described. C.O.C.

### Electron Microscope Studies of the Processes in Leather Manufacture. I—Preliminary Study of the Structure of Leather

D. Burton, R. Reed, and M. J. Wood

*J. Soc. Leather Trades Chem.*, 40, 91-99 (March 1956)

The fine structure of a leather depends upon the mode of tanning; nine electron micrographs are discussed in this connection. The electron microscope should prove useful in studying the mechanism of tanning and also, possibly, in classifying leathers of historical interest. Tanning with Cr is more mild and uniform in effect on the collagen fibrils than is that with Zr salts. Compared with those from full chrome leather, the fibrils of a chrome retan are slightly more full and the spaces between them are filled with large dense particles. Vegetable tanning causes marked modification of the collagen fibrils. The most striking feature of a vegetable-tanned leather is the presence of large dense particles between the fibrils; these appear to be formed by the aggregation of smaller particles and may be insol. tannin derived from the original liquor. Fullness in leathers appears to be associated with broad fibrils of collagen and the presence of much dense particulate matter in the interfibrillary spaces. Oil tanning results in a high proportion of twisted fibrils (this may be a unique feature of oil tanning) similar to those found in a limed pelt; since oil tanning is carried out at a pH value much nearer to that of liming than are the other types, it is possible that the twisted fibrils may originate in the liming process. J.W.D.

### Prevention of Microbiological Deterioration of Leather

S. Dahl

*J. Amer. Leather Chem. Assoc.*, 51, 103-118 (March 1956)

The microbiological deterioration of leather must be prevented when the hide is freshly flayed, since it is impossible to compensate for the destruction of any collagen by bacteria before tanning. The risk of microbiological attack and the methods of prevention at each stage before and during tanning are briefly discussed. Although tanning renders collagen resistant to microorganisms, leather adjuncts such as oils, greases, and even vegetable tannins are excellent nutrients for moulds, especially *Aspergillus niger*. The removal of these adjuncts by bacterial attack renders the leather stiff and brittle; fungicides are used to avert this damage. Methods for the evaluation of this protection are described. The most commonly used fungicide is *p*-nitrophenol, but this is quite easily removed by leaching, and satisfactory alternatives are being sought. The effectiveness of various cpd. as anti-mildew agents is indicated in five tables. Most of the effective cpd. are phenols, chlorine being most advantageous when subst. in the *para* position; the 4-nitro- and 4-thioxyano-phenols are particularly efficient, probably because they are able to combine with the leather by hydrogen bonding. Alkylation of the phenolic cpd. reduces their fungicidal activity. J.W.D.

### Laboratory and Field Exposure Studies of Leather Fungicides

S. Dahl and A. M. Kaplan

*J. Amer. Leather Chem. Assoc.*, 51, 118-136 (March 1956)

The effectiveness of seven experimental leather fungicides (previously evaluated in laboratory tests), in comparison with *p*-nitrophenol, has been studied under field conditions. The field exposure study is reported in considerable detail. Mildew growth under tropical jungle conditions is prevented by *p*-nitrophenol, *o*-chloro-*p*-nitrophenol,

*p*-thiocyanophenol, and bis-*p*-nitrophenyl carbonate, in concn.  $\geq 0.6\%$ . They are adequately stable under severe weathering conditions. *N*-(Trichloromethylthio)tetrahydrophthalimide and tetrachloroquinol inhibit mildew growth at 0.6% concn. in some cases, but they are not reliable. Even at concn.  $\geq 1\%$ , *o*-phenylphenol and 4-chloro-*m*-xyleneol are ineffective. Correlation between the results of field evaluation and laboratory screening tests shows that a failure in the latter will be reproduced in the former, whereas it cannot be assumed that a fungicide that passes the screening test will also pass the field test. J.W.D.

### Capacity of Proteins to bind Cupric Ions

G. H. Wolff and S. Fallab

*Helv. Chim. Acta*, 39, 837-840 (May 1956)

A colorimetric method depending on equilibration with dipyriddyferrous ions is used. For gelatin a relation is established between the degree of its hydrolysis and the amount of copper bound. H.E.N.

### Synthesis of Polycysteine

S. Sakakibara and H. Tani

*Bull. Chem. Soc. Japan*, 29, 85-88 (Jan. 1956)

L-Cysteine and benzenesulphonyl chloride give *S*-thiophenyl-L-cysteine, which, by the action of phosphene, yields the corresponding *N*-carboxy anhydride. Heating this in ethyl thioglycolate causes simultaneous polymerisation and reduction, with the formation of a polycysteine of D.P. ca. 36. Infrared spectra are given. A.J.

### X-Ray Studies of Poly-L-alanine

L. Brown and I. F. Trotter

*Trans. Faraday Soc.*, 52, 537-548 (April 1956)

Completely satisfactory structures have not been obtained for either the  $\alpha$  or the  $\beta$  form of poly-L-alanine, but considerable progress has been made and an outline of the present position is given. Results obtained with other polypeptides are briefly referred to, and a comparison is made with the various silk structures. W.R.M.

### Trypsin-catalysed Reactions of Lysyllysine Ethyl Ester

J. Watson

*Trans. Faraday Soc.*, 52, 592-605 (April 1956)

Measurements are made of the rate of hydrolysis in the presence of trypsin at pH values between 5.6 and 7.6. The results can be explained on the assumptions that only the form of the substrate possessing an uncharged  $\alpha$ -amino group unites with the enzyme to form a complex and that the complex hydrolyses at a rate independent of pH. Trypsin-catalysed reactions of dilysine ester with tyrosine amide and phenylalanine amide are also studied, and it is shown that these amides are of the order of  $10^3$  times as effective as water in decomposing the enzyme-substrate complex. W.R.M.

### Liquid-crystalline Structures in Solutions of a Polypeptide

C. Robinson

*Trans. Faraday Soc.*, 52, 571-592 (April 1956)

Birefringent solutions of poly- $\gamma$ -benzyl-L-glutamate are studied. These show parallel equidistant lines visible in natural light. The periodicity of these lines may exceed 100,000 Å. The structure is associated with very high optical rotatory power, similar to that of the cholesteric type of liquid crystals, but there are some features which have not been observed with the cholesteric type. A model of the structure is tentatively advanced. W.R.M.

Problem of Water in the Manufacture of Leather (II p. 385)  
Ultrasonic Tanning and Dyeing (VIII p. 399)

## XIII—RUBBER; RESINS; PLASTICS

### Macromolecular Compounds

#### LXXXVII—Two-component Polyamides in which one Component is an Amino Acid

V. V. Korshak and T. M. Franze

*Izvestiya Akad. Nauk S.S.S.R.*

*Otdel. khim. nauk*, 98-102 (Jan. 1956)

Previous investigations on two-component polyamides are extended to systems in which one component is 7-aminoheptanoic, 9-aminononanoic, or 11-aminoundecanoic acid, and the other is hexamethylenediamine adipate, azela-ate, or sebacate (cf. *J.S.D.C.*, 71, 479 (1955)).



### LXXXVIII—Polyamides containing Ether Linkages in their Macromolecular Chains

V. V. Korshak and G. N. Chelnokova

*Ibid.*, 103-107

Polyamides containing ether linkages are prepared by reaction of diglycolic acid with hexamethylenediamine and of 3,3'-ethylenedioxybispropylamine with adipic, azelaic, or sebacic acid. Some mixed polyamides are made by the addition of further components to these mixtures. It is found that the introduction of ether oxygens results in lower m.p. and higher solubility, as compared with a polyamide having the same length of repeating unit but not containing ether linkages. It is found also that an ether oxygen in the dicarboxylic acid residue brings about a much greater lowering of the m.p. of the polyamide than an ether oxygen in the diamine residue.

### LXXXIX—Three-component Mixed Polyamide Systems having Amino-acid Components

V. V. Korshak, T. M. Frunze, and T. A. Dikareva

*Ibid.*, 108-113

Details are given of the second part of an investigation on multicomponent polyamides. The essential results of this investigation have already been reported (cf. *J.S.D.C.*, 72, 198 (April 1956)).

### XCIII—Properties of Polyesters of Butane-1:3- and -1:4-diols. XCIV—Polyesters of Propane-1:3-diol and Pentane-1:5-diol

V. V. Korshak and S. V. Vinogradova

*J. Gen. Chem. U.S.S.R.*,

26, 539-544, 544-548 (Feb. 1956)

Work reported previously (see *J.S.D.C.*, 71, 203 (1955)) is supplemented by work on polyesters formed by the title glycols with the first nine of the series of alkanedioic acids. The results are in general accord with the previous observations.

A.E.S.

### Residual Entropy of Linear Polymers

H. N. V. Temperley

*Bur. Stand. J. Res.*, 56, 55-66 (Feb. 1956)

Research Paper 2648

The question of assigning a finite entropy to a polymer is discussed. It is shown that the entropy of chain configurations is the only important contribution at low temperatures. A generalization of the "flexible chain" model is given which is in good agreement with experimental specific heats of polymers and enables a finite entropy to be defined and calculated. Comparison with experimental results throws light on the length of the "effective segment" of polymer chains of various types.

W.R.M.

### Molecular Weights of Thermally Degraded Polymethyl Methacrylate

V. E. Hart

*Bur. Stand. J. Res.*, 56, 67-71 (Feb. 1956)

Research Paper 2649

Molecular weights of residues from thermal degradation in a vacuum of two polymethyl methacrylate samples are obtained at various temperatures. A linear relationship, dependent on temperature, is found between the logarithm of the ratio of residue weight to original weight and the reciprocal of the weight-average mol. wt. Results are explained on the basis of a free-radical chain mechanism.

W.R.M.

### Direct Measurement of the State of Complex Ions adsorbed on Ion-exchange Resins

M. Fujimoto

*Bull. Chem. Soc. Japan*, 29, 285-287 (March 1956)

Studies are made of the absorption of light by suspensions of a basic ion-exchange resin and of resin membranes which have adsorbed a cobalt thiocyanate complex from aqueous solution. Comparison of absorption spectra with those obtained using organic solutions of the complex shows a "red shift" of about 10 mμ.

W.R.M.

### PATENT

#### Leather Substitute

DuP

USP 2,715,588

A porous leather substitute consists of matted inverted fibres of a synthetic linear condensation polymer (40-85% by wt.), a chemically dissimilar incompatible polymeric binder of m.p. < softening point of the fibres (60-15), and void spaces < 3000 (wt. % fibre)<sup>-1</sup> - 30 and > 70%.

C.O.C.

### Effect of Pigments on the Curing of Silicone Resins (IV p. 389)

Process of Formation of Polyamide Resins. V—Rôle of Polymerisation and Polycondensation Reactions in the Process of Converting 6-Hexanolactam into Polymer (VI p. 397)

## XIV—ANALYSIS; TESTING; APPARATUS

### Microtitration of Organic Compounds. IV—Microburette of Constant Rate of Delivery for Automatic Titrations

W. Simon

*Helv. Chim. Acta*, 39, 883-888 (May 1956)

### Polarographic Cell Assembly for Amperometric Titrations

J. P. E. Human and S. J. Leach

*Chem. and Ind.*, 149-150 (3 March 1956)

A cell is fully described, with three diagrams, which has been used in conjunction with a Tinsley polarograph (type 15 5), for the determination of cystine in protein hydrolysates by reduction with Na<sub>2</sub>SO<sub>3</sub> followed by amperometric titration with HgCl<sub>2</sub>.

J.W.D.

### Spectrophotometric Determination of Magnesium with Sodium 1-Azo-2-hydroxy-3-(2:4-dimethylcarboxanilido)-naphthalene-1'-2-hydroxybenzene-5-sulphonate

C. K. Mann and J. H. Yoe

*Anal. Chem.*, 28, 202-205 (Feb. 1956)

The developed colour is measured spectrophotometrically at 510 mμ. The method is sensitive to 1 part of Mg in 50,000,000 parts of soln., and it is applicable to 0.5-10 μg. of Mg. The interferences of various ions are reported, and methods (based upon extraction and upon ion-exchange fractionation) described for the elimination of these.

J.W.D.

### Chelate Complexes and the Chelatometric Determination of Calcium and Magnesium Ions

L. Villa

*Bull. Assoc. Française Chim. Ind. Cuir*,

18, 33-59 (Feb.-March 1956)

A useful review of chelating agents in general, with particular reference to the determination of Ca and Mg.

J.W.D.

### Measurement of Lime-soap Dispersion

P. Beeher

*J. Amer. Oil Chem. Soc.*, 33, 113-116 (March 1956)

A new titrimetric method for the measurement of lime-soap dispersion is described. The method may be used for measuring lime-soap dispersion of formulations over a range of water hardness.

P.G.M.

### Relative Efficiency of Chelating Agents

A. C. Signore, G. M. Gantz, and M. E. Chiddix

*Amer. Dyestuff Rep.*, 45, P 217-P 222 (9 April 1956)

Simple laboratory tests are described for comparing chelating efficiency of different agents, under conditions simulating actual textile operations. Neither the complexing power (measured by the stability constant) nor the complexing capacity (measured by titration with Ca) is a satisfactory criterion of efficiency. Emphasis is placed on control of copper in peroxide solutions and control of iron in alkaline liquors. Loss of peroxide and loss of Ca-complexing capacity at elevated temp. are used, and it is shown that ethylenediaminetetra-acetic acid is better for controlling Cu than N-hydroxyethylethylenediaminetriacetic acid. The latter is, however, superior in controlling Fe in a soda-ash bath. A number of tests are made on the take-up of Fe by different forms of cellulose, viz. cotton flock, rayon flock, and wood pulp, in presence and in absence of complexing agent, but it is concluded that the filter-paper reflectance test is best by virtue of the higher degree of discoloration. A balanced chelating agent is described which is effective over a wide range of processing conditions.

J.W.B.

### Determination of Anionic Detergents in Surface Waters and Sewage with Methyl Green

W. A. Moore and R. A. Kolbenson

*Anal. Chem.*, 28, 161-164 (Feb. 1956)

A procedure is described which is a marked improvement upon that based on Methylene Blue. Interferences from CNS<sup>-</sup>, NO<sub>2</sub><sup>-</sup>, and NO<sub>3</sub><sup>-</sup> are greatly reduced, and the determination is not affected by high concn. of SO<sub>4</sub><sup>2-</sup>, Cl<sup>-</sup>,

$\text{SO}_3^{2-}$ , and  $\text{PO}_4^{3-}$ . The method yields more consistent results than that based upon Methylene Blue. The developed colour is measured spectrophotometrically at 615  $\mu\text{m}$ , max. absorbance being attained at this wavelength irrespective of the identity of the anionic detergent concerned. J.W.D.

#### Paper-chromatographic Procedure for Separating 1-Mono-, 1:3-Di-, and Tri-glycerides, Cholesterol, and Cholesteryl Esters

J. W. Dieckert and R. Reiser

*J. Amer. Oil Chem. Soc.*, **33**, 123-126 (March 1956)

A reproducible paper-chromatographic procedure has been developed for resolving a mixture consisting of 1-mono-, 1:3-di-, and tri-glycerides, cholesterol, and cholesteryl esters. The separation is carried out on silicic-acid-impregnated glass-fibre filter-paper using ethyl ether-*iso*-octane mixtures as the solvent systems. The position of each component may be found by treating with 1:1 sulphuric acid and gently heating the chromatogram. P.G.M.

#### *p*-Phenylazobenzoyl Chloride for Identification and Chromatographic Separation of Colourless Compounds. II—Amines

E. O. Woolfolk and E. H. Roberts

*J. Org. Chem.*, **21**, 436-438 (April 1956)

Aliphatic and aromatic primary and secondary amines have been used, yields ranging from 32 to 85%. Many pairs of cpd. can be resolved by chromatography on alumina using benzene with up to 5% ethyl acetate. H.E.N.

#### Ultraviolet Spectrophotometric Determination of Phosgene with Aniline

W. B. Crummett

*Anal. Chem.*, **28**, 410-412 (March 1956)

Phosgene reacts with aniline in aq. soln. to yield *o*-diphenylurea; this is determined spectrophotometrically at 254.5  $\mu\text{m}$ . There is a sketch of the apparatus. J.W.D.

#### Coal-tar Colours. XX—FD&C Blue No. 2

J. H. Jones, L. S. Harrow, and K. S. Heine

*J. Assoc. Off. Agric. Chem.*, **38**, 949-977 (Nov. 1955)

Methods for the preparation of the isatin-4, 5, 6, and 7-sulphononic acids and the four monosulphonated anthranilic acids are described. These compounds were prepared for use as intermediates for the synthesis of the isomeric indigodisulphononic acids and as reference compounds in studies on the oxidative degradation of the indigosulphononic acids. Isatin-5-sulphononic acid, prepared from sulphanilic acid by the Sandmeyer synthesis, is identical with the compound produced by the sulphonation of isatin and by the oxidation of disulphonated indigo. Spectrophotometric determination of the pure dye content of FD&C Blue No. 2 is the most suitable procedure for routine use. The ultraviolet, visible, and infrared spectra of many of the compounds prepared are given. P.G.M.

#### Chromatographic Procedures for the Separation of Water-soluble Acid Dye Mixtures

R. N. Solar and K. A. Freeman

*J. Assoc. Off. Agric. Chem.*, **38**, 796-809 (Aug. 1955)

The methods of separating 14 water-soluble coal-tar dyes using paper and column chromatography are given. By the latter method sufficient quantities of dyes for spectrophotometric analysis may be separated. P.G.M.

#### Triphenylmethane Dyes derived from Ethylbenzyl-anilinesulphonic Acid

J. H. Jones, M. Dolinsky, L. S. Harrow, K. S. Heine, and M. C. Staves

*J. Assoc. Off. Agric. Chem.*, **38**, 977-1010 (Nov. 1955)

Some of the leuco compounds of FD&C Blue No. 1, Green No. 1, and Violet No. 1 have been prepared and examined. The absorption spectra of various isomeric compounds which constitute the dyes are given and also methods for their spectrophotometric determination. P.G.M.

#### Periodate Test for the Catechol and Quinol Structures

O. M. Windrath

*Anal. Chem.*, **28**, 263 (Feb. 1956)

Catechol or quinol structures may be detected in relatively complex molecules by the addition of 0.25 m. aq. Na metaperiodate to a soln. of the substance under test in methanol; a cryst. ppt. forms within 30 min. in the presence of these structures. Results obtained with 18

cpd. are tabulated and discussed. The test should be used in conjunction with elemental analysis and functional group determination, since similarly placed  $\text{NH}_2$  groups give identical results. J.W.D.

#### Spot Test for Diketones and Quinones based on a Catalytic Effect

F. Feigl and C. C. Neto

*Anal. Chem.*, **28**, 397-399 (March 1956)

1:2-Diketones and quinones catalyse the slow reaction between  $\text{HCHO}$  and *o*-dinitrobenzene to form a violet alkali-metal salt of the aci-form of *o*-nitrosonitrobenzene. Reaction conditions are detailed under which it is possible to detect microgram quantities of the catalytically active substances. The sensitivity of response of 12 cpd. is indicated. Anthracene and phenanthrene may be converted, by evaporation with conc.  $\text{HNO}_3$ , to anthraquinone and phenanthraquinone respectively; these latter cpd. may be detected by the above reaction. Inositol may be detected similarly by conversion to rhodizonic acid; reducing sugars interfere, and modification is necessary when they are present. J.W.D.

#### Colour Tests for Quinones

H. Karius and G. E. Mapstone

*Chem. and Ind.*, 266-267 (21 April 1956)

Quinones may be detected by the colour developed when they react with indole or pyrrole in acid soln. to yield indolylquinones, and by their colour reaction with ethylenediamine. The last method may be applied in either aq. or ligroin soln., or as a spot test on paper; the mechanism is unknown. The colour reactions of several quinones are listed; the sensitivities of the three tests for six quinones are also given. The ethylenediamine test has a limiting sensitivity of 50 p.p.m. as a spot test or in ligroin soln., and of 5 p.p.m. in aq. soln. Although the ethylenediamine test is specific for quinones or quinone-forming cpd., it is not given by anthraquinone. The other two tests are less specific, but amino-subst. quinones do not react, and benzaldehyde interferes under some conditions. J.W.D.

#### Colorimetric Determination of *p*-Acetophenetidide

F. J. Bandelin and R. E. Pankratz

*Anal. Chem.*, **28**, 218-221 (Feb. 1956)

*p*-Acetophenetidide is hydrolysed with acid, and the resulting *p*-phenetidine is diazotised and coupled with  $\beta$ -naphthol in alkaline soln. to give an orange-red dye. The colour may be determined spectrophotometrically at 470  $\mu\text{m}$ . J.W.D.

#### Detection of Dyes possessing a *p*-Phenylenediamine or a *p*-Nitroaniline Structure by means of Spot Tests

F. Feigl and C. C. Neto

*J.S.D.C.*, **72**, 239-240 (May 1956)

When Bandrowski's base and azo dyes containing a *p*-phenylenediamine or a *p*-nitroaniline structure are fused with a dry mixture of sodium formate and sodium hydroxide at 210-230°C. *p*-phenylenediamine is formed by reductive cleavage. Under these conditions the diamine sublimes and the vapour reacts with an acetic-acid solution of aniline and alkali-metal persulphate to yield the indamine dye Phenylene Blue. This procedure may be conducted by the technique of spot test analysis and serves to detect such dyes. The detection limits are in the range 5-10  $\mu\text{g}$ ., as has been verified by testing thirteen pertinent compounds. AUTAORS

#### Spectrophotometric Study of Colloidal Solutions of Dyes

L. I. Belen'kii

*Izvest. Akad. Nauk S.S.S.R., Ser. Fiz.*, **17**, 756 (1953);

*Chem. Abs.*, **50**, 5404 (25 April 1956)

Study of the absorption spectra of colloidal suspensions of polycycloketone, indigoid, sulphur and insoluble azo dyes show that suspensions obey the Bouguer-Lambert-Beer law as do molecular solutions, and that their absorption spectra can be used to characterise the insoluble dyes qualitatively and quantitatively. C.O.C.

#### Analysis of Lipsticks

J. E. Clements

*J. Assoc. Off. Agric. Chem.*, **38**, 838-843 (1955);

*Chem. Abs.*, **50**, 6752 (10 May 1956)

A method of separating the more common lipstick components into the following groups: lakes and fillers,

hydrocarbons, ricinoleic esters, waxes, and fluorescein dyes. C.O.C.

### Detecting and Identifying Oil-soluble Coal-tar Dyes in Food

W. Lindberg

*Z. Lebensm.-Untersuch. und -Forsch.*, **103**, 1-14 (1956); *Chem. Abs.*, **50**, 6096 (10 May 1956)

The dye is extracted with petroleum ether and is in turn extracted from this solution with acid (20 ml. HCl + 10 ml. water made up to 100 ml. with acetic acid or 40 ml. H<sub>2</sub>SO<sub>4</sub> + 10 ml. water and 90 ml. acetic acid), the dye from the acid extract is extracted with ether, the ether evaporated, the residue saponified, the unsaponified material extracted with ethyl acetate and the dye in the extract identified by chromatography on paper impregnated with medicinal paraffin. The solvent used was 80 vol. % methyl alcohol with addition of 5% acetic acid. The R<sub>f</sub> values of Ceres Orange GN, Yellow AB, Yellow OB, Dimethyl Yellow, Ceres Red G, Organol Orange A, Orange SS, Oil Red XO and Organol Red IV for chromatograms in absence and presence of 5 and 10% acetic acid are given in tables. C.O.C.

### Identification of Vat Dyes

D. A. Derrett-Smith and J. Gray

*J.S.D.C.*, **72**, 211-227 (May 1956)

The tables published in 1940 by Derrett-Smith and Bradley (*ibid.*, **56**, 47) and in 1947 by Derrett-Smith and Gee (*ibid.*, **63**, 401) have been brought up to date to include all the British, German, and Swiss vat and solubilised vat dyes which have appeared on the British market since 1940. Dyes which were included in the original tables but have since been made by other firms are included under their new names. In addition, it is shown that certain vat dyes behave differently when treated with sodium hydrosulphite and sodium hydroxide as compared with sodium hydrosulphite and sodium carbonate. AUTHORS

### Changing Frontiers in the Analytical Chemistry of Paint Materials

C. Whalley

*J. Oil & Col. Chem. Assoc.*, **39**, 193-211 (March 1956)

A review of new analytical techniques with special reference to the examination of paint materials; these include microchemical methods, paper and vapour-phase chromatography, and the use of ion-exchange resins and chelating agents. The value of these techniques is illustrated by analytical procedures for various components of the paint system. J.W.D.

### Use of Contrast Ratio in the Measurement of Hiding Power

D. Touth

*J. Oil & Col. Chem. Assoc.*, **39**, 169-184 (March 1956)

For a wide range of pigments of different colours there is good correlation between contrast-ratio determinations and those made by a conventional hiding-power method. Contrast ratio was determined from the ratio of the luminances (or Y values) of the thin paint film when backed by black (0%) and by white (100%). For the method used, the contrast-ratio determination on a thin film is a useful method for determining hiding power rapidly. Changes in hiding power take place during drying, and whilst a wet-state determination is useful for comparing chemically identical pigments, it is only of limited use for comparing chemically different pigments or, more particularly, for comparing paints prepared from different mixtures of pigments. A measurement in the dry state relates more closely to the visual effect as seen by the user of the paint. Contrast-ratio values as determined by the D.E.F. 1053 Method 12 of a series of pigments in paint systems are given, together with an indication of which pigments and which mixtures of pigments satisfy the terms of this specification. The D.E.F. method does not necessarily give results which agree with a visual determination. J.W.D.

### Relationship between Gloss and Surface Roughness of Paint Films

M. Hess

*J. Oil & Col. Chem. Assoc.*, **39**, 185-192 (March 1956)

An instrument, the "Profilometer", may be used to obtain a quant. measure of the roughness (rugosity) of a paint surface. A fine tracer point (radius 0.0005 in.)

constantly weighted with 1.5 g. automatically traverses the surface under examination; the up-and-down movement of the point is transmitted to a coil which moves in the field of a permanent magnet, thus producing a small fluctuating voltage related to the height of the surface irregularities, and this is fed to an amplimeter, where it is amplified and integrated to actuate a direct-reading meter. The instrument is fully described, together with a photograph and a diagram of the tracer head; it is commercially available in Britain. There is an accurately measurable relationship between gloss and rugosity of paint films, and gloss comparisons are made possible (with opaque films) between finishes on, say, a curved surface and those on plane surfaces. Suggestions are made regarding the use of the instrument in studying problems relating to flotation, orange peeling, polishing, colour matching, weathering, and fineness of grind. J.W.D.

### Infrared Analysis of Paint Vehicles based on Alkyd-Nitrogen Resin Blends

C. D. Miller and O. D. Shreve

*Anal. Chem.*, **28**, 200-201 (Feb. 1956)

The three components of a paint vehicle containing urea-formaldehyde, melamine-formaldehyde, and an alkyd resin may be separately determined by a method based upon infrared absorbance measurements at 5.8, 6.1, and 12.25  $\mu$ . on thin films of the vehicle. Calculations are based on absorbance ratios, and control of film thickness is thus unnecessary. J.W.D.

### Instrument for Measuring the Permanent Elasticity and Adhesion of Coloured Films

W. Pragel

*Farbe und Lack*, **62**, 59-62 (1956);

*Chem. Abs.*, **50**, 6809 (10 May 1956)

Paint or lacquer is applied to a rubber balloon mounted on a support similar to that used for a terrestrial globe. The balloon is connected to a small rubber bellows and a hand bulb through rubber tubing and a T joint. The balloon is inflated by squeezing the hand bulb and a closed system established between the balloon and the bellows by closing that branch of the T joint connected to the hand bulb. The coated balloon is repeatedly inflated and deflated by compressing and expanding the bellows which are mechanically driven. More than one sample of paint can be applied to the balloon. The variation in the volume of the balloon can be made small or great by varying the length of the drive rods for the bellows. A marker indicates the number of inflation-deflation cycles. This machine enables permanent elasticity to be measured, whereas the Erickson test allows only a single extension of the film. C.O.C.

### Identification of Inks by Paper Chromatography

S. Iwasaki

*Science & Crime Detection* (Japan), **8**, (3), 36 (1955)

### Identification of Textile Fibres by Dye Staining Techniques

J. D. Donovan, J. A. Beegan, and G. H. Hummel

*Text. Research J.*, **26**, 227-228 (March 1956)

Du Pont Identification Stain No. 4 is effective in distinguishing and identifying the following textile fibres in mixtures containing any or all of them—cellulose acetate (stained reddish orange), cotton, cuprammonium rayon, viscose rayon (shades of blue and green), wool, Vicara, nylon (shades of purple-brown), Acrilan (reddish brown), dynel (brown), Orlon 81 (grey-blue), Dacron and Orlon 42 (shades of yellow-orange). Dacron and Orlon 42, which give similar colours, may be further distinguished by boiling in 5% H<sub>2</sub>SO<sub>4</sub>, when Orlon 42 becomes pink. Other confirmatory tests are given. J.C.F.

### Instrumental Ranking of Fabric Handle. IV—Ranking of Various Fabrics by Withdrawal Method in Comparison with Other Methods

H. Uchida, T. Takizawa, and M. Kobayashi

*J. Soc. Textile Cellulose Ind. Japan*, **12**, 288-293 (April 1956)

### Behaviour of Flax, Hemp, and Ramie in Cuprammonium Solution

H. Klenk

*Textil Praxis*, **10**, 1202-1204 (Dec. 1955)

The main distinguishing features observed under the microscope after treatment of these fibres with cuprammonium solution are—flax shows threads of protoplasm; with hemp the middle lamella collapses and forms



a finely creased band; with ramie lateral swelling is very marked, with formation of many cracks in all directions. 15 photomicrographs. C.J.W.H.

#### Complexometric Estimation of Copper in Cuprammonium and Cuprammonium Cellulose Solutions

B. Philipp and H. Hoyme

*Faserforsch. und Textiltech.*, 7, 231-232 (May 1956)  
A method is described in which dilute cuprammonium solution or cuprammonium solution containing cellulose is titrated with disodium ethylenediaminetetra-acetate, murexide being used as indicator. Results agree with those obtained by the electrolytic method. W.R.M.

#### Surface Structure of Wool and its Components revealed by Metal Shadowing

R. D. B. Fraser and G. E. Rogers

*Australian J. Biol. Sci.*, 8, 129-135 (Feb. 1955)  
Gold shadowing reveals considerable details of surface modifications in damaged wool fibres when viewed with transmitted light in the optical microscope. The method is more rapid and less likely to introduce artefacts than the replica techniques currently employed in electron microscopy. New evidence on the nature of the inner surface of the cuticle is also described. P.G.M.

#### Electron-microscope Studies of the Surface Structure of Wool and Other Fibres

N. Ramanathan, J. Sikorski, and H. J. Woods

*Biochim. Biophys. Acta*, 18, 323-340 (Nov. 1955)  
Two methods of obtaining SiO replicas of fibre surfaces are described; both make use of a technique in which the fibre is partly embedded in a suitable plastic, leaving only a fraction of the surface exposed. Both techniques yield replicas showing much more detail than has previously been reported to be present in wool fibre surfaces. The technique permits experimental investigation of the reproducibility of the replicas, and gives the possibility of comparing the same surface area before and after some surface treatment. Interpretation of the micrographs is facilitated by using stereoscopic methods, and in this way several new features of the wool-fibre cuticle have been observed. Micrographs of the surfaces of cotton, jute, cellulose acetate, and raw silk are given. P.G.M.

#### Permanent Set of Wool Fabric

S. Okajima and T. Inoue

#### I—An Experimental Press and Method of Measurement of Lustre of Fabric

*J. Soc. Textile Cellulose Ind. Japan*, 12, 217-221 (March 1956)

A special press was prepared in order to study the crabbing and the pressing of wool fabrics. The pressing surface is  $15 \times 15$  sq.cm., and a sample fabric or fabrics can be subjected to various pressures at the required temperatures. The fabrics can be treated in the wet or the dried state, the former corresponding to a crabbing and the latter to a pressing process in the factory. The setting effect of these treatments is appraised by observing the permanency of the lustre of the fabrics treated.

#### II—Effect of pH of the Crabbing Water upon the Setting Effect

*Ibid.*, 12, 280-287 (April 1956)  
Wool fabrics were immersed in water of pH 7-10.5 at 50°C. for 1 hr., and pressed at 0.5-4 kg./cm.<sup>2</sup> and 100°C. for 5-20 min. immediately after being taken out of the water. The pressing was given to a sheet or a pile of six fabrics. The lustre  $L$  of these fabrics decreases, in general, to  $L'$  when they are released for 10-20 min. in steam at 100°C. This decrease of lustre is less the more effectively the fabric is set by the crabbing. So  $L/L'$  was measured as an appraisal of the effectiveness of the crabbing, and the following results were obtained—(1) The crabbing is most effective when the pH of the water is 9.2, when  $L$  and  $L'/L$  are largest. (2) At pH 8,  $H_2S$  could be smelt during the pressing and the samples became slightly yellowish. This correspondence between evolution of  $H_2S$  and effect of crabbing is interesting from the standpoint of the mechanism of chemical setting.

AUTORS

#### Analysis of Mixtures of Protein and Non-protein Fibres by means of Sodium Hypochlorite

E. Druce

*J. Textile Inst.*, 47,  $\pi$  202- $\pi$  208 (April 1956)  
A 1.0 N. solution of NaOCl dissolves the following fibres completely within 15 min. at room temp.: wool, chlorinated

wool, raw and degummed Bombyx silk, cashmere, mohair, Ardil B and F, Fibrolane BX, Lanital, and soya-bean fibre. Tusseh silk requires slightly longer treatment, but Vicara dissolves only to a slight extent unless NaOH is added to the NaOCl. It is important that there should be no excessive fall in pH during the reaction. Bleached cotton, viscose rayon, Terylene, nylon, Perlon, Orlon, Vinyon, Fibravyl, and polythene do not dissolve appreciably. A method of separation, based on the above facts, is as follows: Fatty matter, size, etc. are removed by appropriate methods. A sample (0.3-0.5 g.) of known regain is accurately weighed, and transferred to a 100-ml. beaker. For protein fibres other than Vicara about 50 ml. of N-NaOCl (the strength is not critical) containing 5 g. of added NaOH per litre is added, and the material well stirred for 15 min. (30 min. for Tusseh). The product is filtered, the beaker washed out with a little NaOCl, and the residue washed with cold water, 0.5% acetic acid, and water, and finally dried for 3 hr. at 110°C. If the protein fibre is Vicara the sample is first treated with 50 ml. of 4% NaOH and left for 30 min. with occasional stirring; it is filtered and washed, and then replaced in the beaker and treated with 50 ml. of alkaline NaOCl as above. A correction factor of 1.03 must be applied if the non-protein component is raw cotton, and of 1.01 for viscose rayon if mixed with Vicara. The procedure is not recommended for blends with cellulose acetate. J.W.B.

#### Colorimetric Ninhydrin Reaction for Measurement of $\alpha$ -Amino Nitrogen

H. Kalant

*Anal. Chem.*, 28, 265-266 (Feb. 1956)  
The photometric ninhydrin reaction of Cocking and Yemm (*Biochem. J.*, 58, xii (1954)) may be applied to the determination of free amino nitrogen in tungstic acid filtrates of plasma. Excess of cyanide reagent or ninhydrin, or the presence of peroxides, interferes. Use of a citrate buffer eliminates interference from metal salts. Other protein precipitants are not satisfactory. J.W.D.

#### Preservation of Ninhydrin-Amino Acid Chromatograms

G. Zweig

*Anal. Chem.*, 28, 428 (March 1956)  
The ninhydrin-treated chromatogram is dipped in dil.  $Cu(NO_3)_2$  (1 ml. of saturated aq.  $Cu(NO_3)_2$  and 0.2 ml. of 10% vol./vol.  $HNO_3$ , diluted to 100 ml. with ethanol). The paper is quickly neutralised with ammonia vapour and air-dried; finally the paper is sprayed with an acrylic aerosol. J.W.D.

#### Titration Method for Estimating the Felting Qualities of Carroted Rabbit Fur

P. Hückel

*Textil Praxis*, 10, 1252-1254 (Dec. 1955)  
Pyridine, HAFRA, alkali solubility, and pH methods for assessing the felting properties of carroted rabbit fur are discussed. The author describes a new method in which the cut fur is treated with hot potassium permanganate, and an aliquot of the solution is treated with oxalic and sulphuric acids and back-titrated with  $KMnO_4$ . B.K.

#### Microscopic Examination of Modified Starches

T. J. Schoch and E. C. Maywald

*Anal. Chem.*, 28, 382-387 (March 1956)  
The tendency of starch granules to aggregate may be measured by examination of a 0.2-0.3% suspension of the starch in water and in glycerol. The presence of adjuncts such as borax or carboxymethylcellulose may be revealed by the use of non-aqueous mountants. The composition of starch blends can often be evaluated by granule counts in a haemocytometer. The species of a pregelatinised starch may be determined by destroying the gelatinised material with an enzyme and examining the ungelatinised residue. Gelatinisation temp. may be simply and accurately measured on a microscope hot-stage, and it can be used as a measure of the extent and the uniformity of chemical modification. Anionic products, such as oxidised starches, phosphate esters, and carboxymethyl ethers, are stained by positively charged dyes such as Methylene Blue; cationic starches stain only with negatively charged dyes such as Light Green SF. Intensity and uniformity of staining yield evidence of blended starches and non-uniform derivatives. There are three photomicrographs, and several graphs and tables of results. J.W.D.



### Colorimetric Method for Determination of Sugars and Related Substances

M. Dubois, K. A. Gillies, J. K. Hamilton, P. A. Rebers, and F. Smith

*Anal. Chem.*, **28**, 350-356 (March 1956)

Simple sugars, oligosaccharides, polysaccharides, and their derivatives (including the methyl ethers with free or potentially free reducing groups) give an orange-yellow colour when treated with phenol and conc.  $H_2SO_4$ ; they may be determined in submicro quantities by measuring the developed colour spectrophotometrically at 490 m $\mu$  for hexoses and 480 m $\mu$  for pentoses and uronic acids. In conjunction with paper partition chromatography the method may be used to determine the composition of polysaccharides and their methyl derivatives. Numerous absorption curves are reproduced and absorption data are tabulated for 30 cpd.

J.W.D.

### Spectrophotometric Method for Quantitative Evaluation of Early Stages of Hydrolysis of Branched Components of Starches by $\alpha$ -Amylase

J. W. Van Dyk and M. L. Caldwell

*Anal. Chem.*, **28**, 318-320 (March 1956)

The initial stages of the hydrolysis of waxy maize starch by pancreatic amylase from swine may be followed by means of a method based upon the absorption spectra of the complexes of iodine with the substrate and with its early hydrolysis products. The method can also be used, under suitable conditions, to follow the early stages of the hydrolysis of the branched components of other starches by pancreatic amylase or by other  $\alpha$ -amylases. By calibration of the absorption ratios against the free CHO groups formed, the velocity of the hydrolysis obtained by the spectrophotometric method may be expressed in terms of the glucosidic linkages hydrolysed.

J.W.D.

### Cellulose Analysis and Bleaching Methods for Cellulosic Materials

S. Honda and M. Okabe

#### VI—New Method for estimating the Degree of Bleaching of Unbleached Chemical Pulps

*J. Soc. Textile Cellulose Ind. Japan*, **12**, 266-269 (April 1956)

The present author has reported in the *Bull. Agricultural Chem. Soc. Japan* a new method of estimation of cellulose by the use of bleaching powder. Continuing the experiments, by a modification of this method the writer has been able to estimate the degree of bleaching of unbleached chemical pulps. The results gained not only explained in detail the chemical reactions of bleaching but also gave the "chlorine numbers" both a theoretical and a practical basis. These procedures are as follows—

A—Each of the acidified chlorinated liquors is filtered through a glass filter, and the filtrates and wash liquors are collected and titrated with 0.1 N- $Na_2S_2O_3$ , giving the chlorine consumed ( $Cl_2$ ).

B—After each chlorinated residue has been boiled with 20%  $Na_2SO_3$  soln., it is filtered off on a glass filter. The filtrates are boiled for 3 hr., and a known volume of 0.1 N- $AgNO_3$  and a quantity of conc.  $HNO_3$  are added. When cool, the reaction mixture is titrated by the Volhard method to determine the soluble Ag. From these titration figures, the combined chlorine ( $Cl_X$ ) is obtained.

In these procedures blank tests should be made to correct the results. The chlorinations should be repeated till the consumption of chlorine reaches zero and likewise the combined chlorine is zero.

The summary of each consumed chlorine ( $\Sigma Cl_2$ ) is the "chlorine number for the bleach requirement of unbleached chemical pulp" by the writer's titration method. Furthermore, it is considered that the total of each combined chlorine is the combined chlorine for the lignin in the unbleached chemical pulps.

AUTHORS

### Microscopical Studies on Wool Dyeing—Preparation and Use of Longitudinal Sections

H. E. Millson

*Amer. Dyestuff Rep.*, **45**, P 331-P 336 (21 May 1956)

A simple method for obtaining longitudinal cross-sections of wool fibres consists in pouring molten paraffin wax flakes into chilled plaster of Paris moulds in which wool fibres have been fastened and, after cooling, cutting sections parallel to the long axis of the fibres with a new razor blade. Sections are suitably mounted, and examined

to find good sections. Good results are obtained with the coarser fibres (further refinement appears necessary for fine types), and photomicrographs are shown of various dyed samples, illustrating dyeing irregularities not normally visible.

J.W.B.

### Instrumental Control in Colour Matching

E. Atherton

*Proc. Tech. Sectn. British Paper & Board Makers' Asscn.*, **36**, 559-586 (Dec. 1955)

Colour measurement is discussed with particular reference to the C.I.E. system. Different types of instruments may be more suitable for specific problems, and, in this connection, possible errors of measurement with spectrophotometers and colorimeters are mentioned. The use of optical instruments for colour matching is briefly outlined; simple filter colorimeters may be of considerable value for charting the properties of ranges of dyes and for indicating the most suitable choices for matching problems.

K.W.

### Machine for testing Rubbing Fastness

K. Fujino

*J. Textile Machinery Soc. Japan*, **1**, 70 (Nov. 1955)

The sample is held over a cylindrical face which rests on a reciprocating arc-shaped bed.

A.J.

### Colour Illusions and Aberrations during Stimulation by Flickering Light

W. G. Walter

*Nature*, **177**, 710 (14 April 1956)

A number of people of normal colour vision were given the Ishihara test by the light of an electronic stroboscope giving blue-white flashes lasting  $\sim 20 \mu\text{sec}$ . and with a peak illumination of  $\sim 80,000$  ft. candles. The flash-rate was set at 1 flash/sec. and the subject was asked to comment on the pattern seen; the flash-rate was gradually increased after each comment. Most subjects found no difficulty with the orange-grey contrasts (cards 1 and 22), but were confused and often mistaken with the brown-green and blue-yellow cards. Several people gave evidence of temporary protanopia or deuteranopia during flicker at  $< 20$  flashes/sec. The relation between aberration of colour vision and flicker frequency was not consistent, but most people were particularly erratic in judgment at frequencies of 5-10 flashes/sec. The combination of normal and colour-blind responses to give an "original" response was commonly observed; e.g. 5 for normals (2 for colour-blind) was seen as 3. The effect cannot be satisfactorily accounted for in terms of existing colour-vision theory.

J.W.D.

### Colour Measurement—Report of the Colour Committee of the Society of Leather Trades' Chemists

J. S. Mudd (Convener)

*J. Soc. Leather Trades Chem.*, **40**, 41-57 (Feb. 1956)

The colour of leather and leather finishes may be measured for record purposes by means of photoelectric surface colorimeters; the Hilger & Watts model J. 20 is recommended as giving the required accuracy without high capital outlay. Full details are given of the evolution of a method for determining a colour tolerance or  $T$  figure, based on liminal differences under normal lighting and viewing conditions as distinct from the refined conditions used in making photometric observations for the purposes of fundamental research. The tolerance allowed for change in chromaticity represents a noticeable difference in the appearance of a pattern, and that allowed for luminance also represents such a difference; it follows that the full magnitude of both these tolerances should not operate at the same time. The combined tolerance  $T$  is given by the formula—

$$T = \frac{\sqrt{c_t^2 + \beta_t^2}}{100}$$

where  $c_t$  is the percentage chromaticity tolerance used, and  $\beta_t$  is the percentage luminance tolerance. A difference between patterns of 1.0  $T$  represents a just perceptible difference when patterns of normal size are viewed under normal lighting conditions, and smaller tolerances are not considered to be useful or necessary in the leather industry. The method can be used for the control of production leathers and finishes, and for the recording of changes due to fading, washing, weathering, ageing, wet or dry rubbing, or chemical action.

J.W.D.

### Determination of the Resistance of Cellulosic Textiles to Damage by Micro-organisms

H. Ostertag

DIN 53,930-53,933

*Textil Praxis*, 11, 572-580 (June 1956):*Faserforsch. und Textiltech.*, 7, 526-529 (July 1956):*Melliand Textilber.*, 37, 708-714 (June 1956)

Standard methods are detailed for the assessment of the resistance of cellulosic materials to attack by fungi and bacteria. Introductory notes on the methods used are included. B.K.

### Burning Tests on Textiles

DIN 53,906 and 53,907

*Textil Praxis*, 11, 470-473 (May 1956)

See J.S.D.C., 72, 367 (July 1956)

### Determination of the Formaldehyde Yield of Cellulose Textiles treated with Formaldehyde, Urea-Formaldehyde, or Melamine-Formaldehyde

W. J. Roff

*J. Textile Inst.*, 47, T 309-T 318 (June 1956)

A sample (0.02 g.) of cellulosic fabric containing a formaldehyde-type finish is treated with 100 ml. of 12N-H<sub>2</sub>SO<sub>4</sub> overnight at 25°C. A sample of the hydrolysis liquor is filtered through sintered glass, and a 5-ml. aliquot transferred to a 6 in. x 1 in. boiling tube. After addition of 1 ml. of 5% chromotropic acid and 5 ml. of conc. H<sub>2</sub>SO<sub>4</sub>, the contents are mixed, and the tube is hung for 30 min. with the lower half submerged in boiling water. The red solution is cooled, transferred to a 50-ml. volumetric flask, and adjusted to volume. The colour intensity is measured at 570 mμ. by means of a spectrophotometer or an absorptiometer, and the concentration of formaldehyde calculated from a previously made calibration curve. An investigation is made of critical factors during hydrolysis, covering adequacy of treatment, presence of cellulose, time, temperature, and acid strength; and of critical factors during the formaldehyde determination. These show that a true value is obtained after 16-24 hr. hydrolysis. J.W.B.

### Determination of Silicones in Textile Materials

G. M. Petty

*Anal. Chem.*, 28, 250-251 (Feb. 1956)

The silicone-containing material is wet-ashed with conc. HNO<sub>3</sub>, H<sub>2</sub>SO<sub>4</sub>, and HClO<sub>4</sub>. The resulting SiO<sub>2</sub> is determined by weighing the crucible before and after treatment of the residue with H<sub>2</sub>SO<sub>4</sub>-HF. The weight of SiO<sub>2</sub> x 1.00 = methyl hydrogen silicone; SiO<sub>2</sub> x 1.23 = dimethyl silicone. J.W.D.

### Evaluation of Viscose Pulps—I

E. Treiber and L. Stockman

*Svensk Papperstidning*, 59, 157-171

(15 March 1956)

The present methods of testing viscose pulps provide insufficient data to predict accurately the characteristics of the rayon to be prepared from viscose, in particular its fibre-technological properties. The literature dealing with the evaluation of various pulp properties has therefore been critically examined in order to find out to what extent these properties, alkali solubility and insolubility, pentosan content, D.P. and polymolecularity, allow the quality and yield of rayon to be characterised. The significance of pulping conditions, the species of wood used, and the purity of the pulp and the fibres are also discussed. K.W.

### Determination of the Alkali Solubility of Pulp

[Swedish] Central Laboratory of the Pulp Industry Analysis Committee. CCA 8:55 (Revised)

*Svensk Papperstidning*, 59, 186-188

(15 March 1956)

The main changes in the revised standard are a lowering of the pulp concentration from 5 g. to 1.5 g. per 100 ml. of solution and an increase in the treatment time from 45 to 60 min. K.W.

### Determination of Total Sulphur in Viscose

H. Demus

*Faserforsch. und Textiltech.*, 7, 268-275

(June 1956)

Various methods of determination are compared and different sources of error considered. Conditions for a reliable volumetric method of estimation are discussed, and an automatic pipette, suitable for serial tests, is described. W.R.M.

### Determination of the Active Agent in Carboxymethylcellulose Preparations

K. Wilson

*Svensk Papperstidning*, 59, 218-222

(31 March 1956)

A comparison of various existing methods for the determination of Na CMC is followed by a description of a method suitable for technical, low-viscosity CMC products according to which the pure Na salt is isolated by combined washing with acidified and neutral ethyl alcohol. K.W.

### Proposed Revised Official Methods for the Analysis of Oils, Fats, and Waxes

D. Burton and G. F. Robertshaw

*J. Soc. Leather Trades Chem.*, 40, 170-177 (May 1956)

### Rapid Method of estimating "Leather Substance"

G. J. Cutbush

*J. Soc. Leather Trades Chem.*, 40, 67-73 (Feb. 1956)

### Vegetable-tanned Leather Analysis

Society of Leather Trades Chemists

*J. Soc. Leather Trades Chem.*, 40, 58-66 (Feb. 1956)

### Chemical Analysis of Chrome-tanned Leathers and Chrome Tanning Materials

Society of Leather Trades Chemists

*J. Soc. Leather Trades Chem.*, 40, 163-169 (May 1956)

### Studies on Leather by means of a Sonic Technique

J. R. Kanagy and M. Robinson

*J. Amer. Leather Chem. Assocn.*, 51, 174-197

(April 1956)

The velocity of sound through leather varies substantially with changes in chemical and physical structure (particularly with fibre orientation), and it may thus be used to reveal modifications of the fibrous order produced by strain, ageing, and filling. The results show good correlation with those of tensile and elongation tests. The effects of tannage, grease, and moisture can also be demonstrated. Hide-to-hide and point-to-point variations are extensive, and several factors influencing velocity simultaneously are not readily distinguishable. The speed of sound increases with the period of ageing at 100°C. to a max. The method has the advantage of being non-destructive. J.W.D.

### Paper-chromatographic Analysis for Collagen and Collagen Derivatives

J. M. Cassel

*J. Amer. Leather Chem. Assocn.*, 51, 223-234

(May 1956)

A chromatographic method is described which consists in a two-dimensional separation on paper together with a spectrophotometric finish after development with ninhydrin. The analysis of collagen by this method agrees well with the generally accepted data in the literature. The results obtained for tyrosine, phenylalanine, and hydroxyproline were checked against and found to agree well with those obtained by specific colorimetric tests for these amino acids. J.W.D.

### Index of Refraction and Particle Size as Factors in the Infrared Spectrophotometry of Polyvinyl Chloride

M. R. Harvey, J. E. Stewart, and B. G. Achhammer

*Bur. Stand. J. Res.*, 56, 225-235 (April 1956):*Research Paper 2670*

### PATENT

### Testing the Draping Properties of a Fabric

Fabric Research Laboratories

USP 2,714,328

A circular sample of the fabric is supported by a flat disc of less diameter than the sample. The amount of drape is then measured by progressively determining the projected horizontal radial distance from the supported central portion of the sample to the draped peripheral edge of the sample. C.O.C.

### Ion-exchange Terminology (II p. 384)

Spectrophotometry of Dyes, I—Methyl Green. II—Pyronine (IV p. 387)

Polarographic Investigation of Fur Dyes and their Oxidation Products (IV p. 388)

Control of Sodium Hydrosulphite in Vat Dyebaths (VIII p. 400)

Capacity of Proteins to bind Cupric Ions (XII p. 406)

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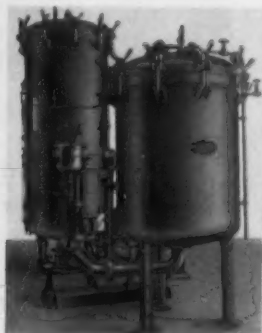
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**CANADA**—Dyehouse Chemist for laboratory of Canadian Celanese Limited, Drummondville, Quebec. Age 25 to 35, preferably married. Must have a sound knowledge of textile chemistry, and be able to organise and control laboratory staff. This position offers a sound opportunity for advancement with an expanding organisation, and carries with it Group Insurance and Superannuation benefits. Interviews will be arranged in England, and passage for the successful applicant will be fully paid. Reply giving full details to Dyehouse Superintendent.

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The Society is urgently wanting Journals for all months of 1955 except July and December. The months of August and September 1955 are particularly required. Copies of January, February, March, and April 1956 issues are also in demand. Please address communications to the General Secretary.

## PERKIN CENTENARY EXHIBITION

LEEDS UNIVERSITY

26-28 SEPTEMBER 1956

The Colour Chemistry and Dyeing Department at the University of Leeds is to hold an Exhibition to commemorate the centenary of the discovery of Mauve by W. H. Perkin, the founder of the synthetic dyestuffs industry. The Exhibition will illustrate the growth of the dyestuffs industry since Perkin's time and recent developments to meet the need for colouring the new man-made fibres. Anyone interested in dyes and dyeing is invited to visit the Exhibition, which will be open from 2 p.m. to 5 p.m. on Wednesday, 9 a.m. to 5 p.m. on Thursday, and 9 a.m. to 4 p.m. on Friday, 26th-28th September.

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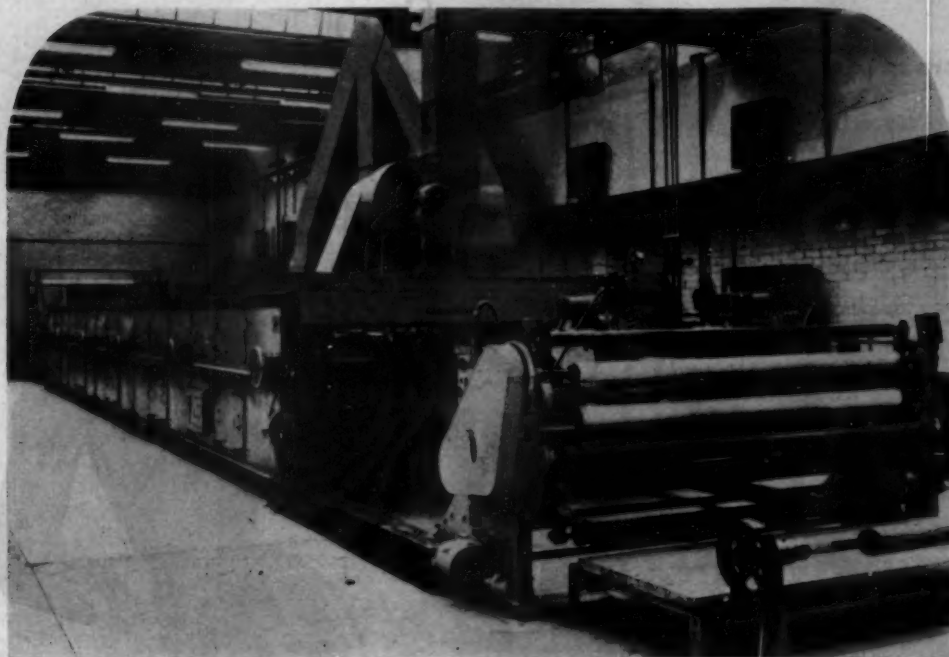
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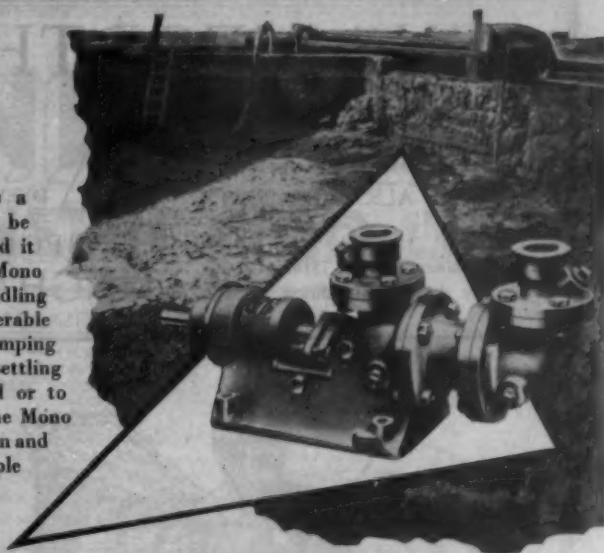
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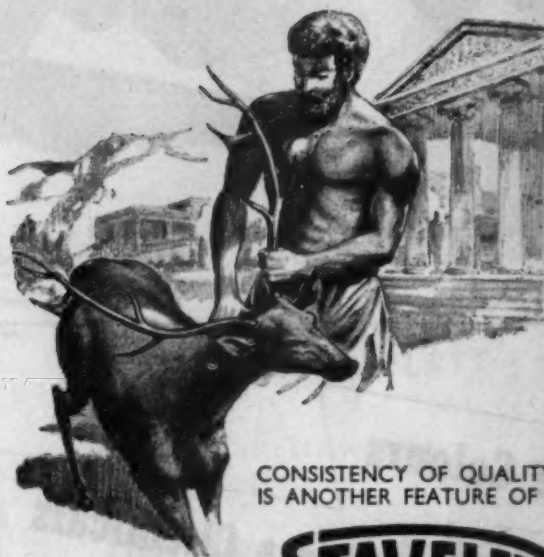


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## INDEX TO ADVERTISERS

| Page    |                                     |
|---------|-------------------------------------|
| xxv     | Alcock (Peroxide) Ltd               |
| xxiv    | Allied Colloids (Mfg) Co Ltd        |
| vi      | Amoa Chemical Co Ltd                |
| xiv     | Badische Anilin- & Soda-Fabrik A G  |
| xxvii   | Bayer Leverkusen Germany            |
| xxxvii  | British Industrial Plastics Ltd     |
| xx      | Brotherton & Co Ltd                 |
| ii      | Brown & Forth Ltd                   |
| xxi     | Ciba Ltd                            |
| xviii   | Clayton Dyestuffs Co Ltd            |
| xxxix   | Cole & Wilson Ltd                   |
| xli     | Colne Vale Dye & Chemical Co Ltd    |
| xix     | E I du Pont de Nemours & Co (Inc)   |
| xxxii   | Farbwerke Hoechst A G               |
| xl      | Chas Forth & Son Ltd                |
| xlii    | Gardinol Chemical Co Ltd            |
| xvi     | Geigy Co Ltd                        |
| xliiii  | Hardman & Holden Ltd                |
| xxiii   | L B Holliday & Co Ltd               |
| xii     | Imperial Chemical Industries Ltd    |
| xxvi    | Imperial Chemical Industries Ltd    |
| vi      | Johnsons of Hendon Ltd              |
| xv      | Laporte Chemicals Ltd               |
| xxxiv   | Longelose Engineering Co Ltd        |
| xxxviii | Mather & Platt Ltd                  |
| xxxix   | Mono Pumps Ltd                      |
| xli     | Newell (Chemicals) Ltd              |
| xliv    | Sir James Farmer Norton & Co Ltd    |
| i       | Nu-Swift Ltd                        |
| xxx     | Samuel Pegg & Son Ltd               |
| ii      | L J Pointing & Son Ltd              |
| xxviii  | James Robinson & Co Ltd             |
| xi      | Sandoz Products Ltd                 |
| xiii    | Sandoz Products Ltd                 |
| vii     | Saunders Valve Co Ltd               |
| ii      | T Saville Whittle Ltd               |
| iv      | Shell Chemical Co Ltd               |
| v       | Shirley Developments Ltd            |
| xxix    | F Smith & Co (Whitworth) Ltd        |
| xl      | Standard Chemical Co                |
| xl      | Staveley Iron & Chemical Co Ltd     |
| xlii    | Stockport United Chemical Co Ltd    |
| xxx     | Joseph Stubbs Ltd                   |
| xxxiii  | Tennants Textile Colours Ltd        |
| viii    | W P Thompson & Co                   |
| xvii    | West Indies Chemical Works Ltd      |
| x       | J B Wilkinson (Chemicals) Ltd       |
| xxii    | Yorkshire Dyeware & Chemical Co Ltd |

